

# Intercalation of Mg–Al layered double hydroxide by anionic surfactants: Preparation and characterization

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Received 17 October 2006; received in revised form 1 March 2007; accepted 7 March 2007

Available online 23 March 2007

## Abstract

The preparation and the characterization of intercalation compounds of Mg–Al layered double hydroxide (Mg–Al LDH) and various organic surfactants have been studied in detail. The preparation has been carried out following regeneration method. The aim of such organic modification is to prepare LDHs suitable for application in polymer-LDH nanocomposites. The LDH-surfactant hybrids have been characterized by wide angle X-ray scattering (WAXS) and Fourier transform infrared (FTIR) spectroscopy. The modified Mg–Al LDH materials show an increase in interlayer distance as compared to the unmodified Mg–Al LDH depending on the length of the surfactant anions. Thermal decomposition changed significantly after organic modification. The particle morphology was investigated by scanning electron microscopy (SEM).

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**Keywords:** Layered double hydroxide; Intercalation compounds

## 1. Introduction

The layered double hydroxides (LDHs) are layered crystalline materials well known for their applications in the field of heterogeneous catalysis (Cavani et al., 1991; Tichit and Coq, 2003). Other fields of application include the usage as heat stabilizer (Van Der Ven et al., 2000), as molecular sieve or ion exchanger (Miyata, 1983), in designing of sensors and biosensors based on clay modified electrodes (Mousty, 2004), as halogen scavengers (Choy et al., 2001), in biomedical applications (Cavani et al., 1991; Wang and Zhang, 2004), etc. Recently, there has been a growing interest in using

these materials for clay–polymer nanocomposite synthesis (Leroux and Besse, 2003; Hsueh and Chen, 2003a,b; Chen and Qu, 2003, pp. 3208–3213; Liao and Ye, 2003; Costa et al., 2005). Our interest in Mg–Al LDH especially concerns its application in polymer nanocomposite synthesis. The reason behind studying LDH materials as potential nanofillers for polymer is not just that it could be treated like other nanofillers (such as layered silicates) for the same purpose. But, LDHs present certain specific advantages, which are lacking in layered silicates type nanoclays and are listed below.

- LDHs are reactive materials that undergo endothermic decomposition producing water and metal oxide residue. This property could be useful in improving the thermal stability and flame-retardancy of polymer matrix more efficiently (Qiu et al., 2006).

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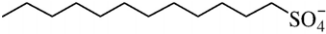
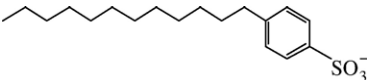

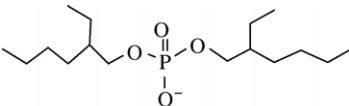
- b. Unlike cationic clays (like layered silicates, which are usually modified by organic amines), LDHs could be modified with anionic surfactants. This is certainly of a great importance, if the wide range of choice for surfactants, like fatty acid salts, sulfonates, sulfates, phosphates, *etc.*, is considered (Carlino, 1997; Meyn et al., 1990; Prevot et al., 2001; You et al., 2002a, 2002b).
- c. In clay–polymer nanocomposites, the reinforcing efficiency of the nanoclay depends on its extent of exfoliation (*i.e.* the number of delaminated single crystal layer or their small stacks) in the polymer matrix. A fully exfoliated nanocomposite based on LDH would contain more number of exfoliated layers per unit amount of filler added than the layered silicate–polymer nanocomposites. This is due to the fact that each crystal layer of LDHs is composed of single octahedral sheet of mixed metal hydroxide whereas that in layered silicate type clays is composed of multiple octahedral/tetrahedral sheets of metal oxide/hydroxide. Therefore, principally, LDHs will provide more efficient reinforcement of the polymer matrix at lower filler concentration.
- d. LDHs could be synthesized in laboratory with high chemical purity, homogeneous structure and tunable chemical compositions.

For the synthesis of polymer nanocomposites, pristine LDH is not suitable as it has very small interlayer spacing, which makes intercalation of polymeric chains/segments practically impossible. Therefore, pre-treatment of LDH is necessary. This pre-treatment often involves the insertion of organic anion species having a long hydrophobic tail, which on

one hand causes the expansion of the interlayer distance and on the other makes the LDH materials more compatible with organic polymers. The aim of the present study is the preparation of Mg–Al LDH-surfactant hybrids using anionic surfactants having different functionalities and varying hydrocarbon tail lengths followed by the characterization of the hybrids. There are several ways reported in literature for modification of LDH, for example co-precipitation in the presence of organic species, ion exchange method, regeneration method, *etc.* (Carlino, 1997; Newman and Jones, 1998; Bergaya et al., 2006). Among these, the regeneration method has been found to be more effective, especially with LDH containing carbonate anion (You et al., 2002a,b). Another advantage of the regeneration method is the absence of or a minimum concentration of competing anions, carbonate in this case.

The regeneration method for modification of LDH is based on a unique property of these materials. Most of the LDH clays can regenerate their original structure from their oxide form, when the latter is dispersed in an aqueous solution containing the anion present in the original material. This behavior is also confirmed for the Mg–Al LDH synthesized for the present study. It shows recovery of the crystal structure, though there occurs some loss in the degree of crystallinity. To obtain the oxide form suitable for regeneration process, the pristine LDH is heated at 450 °C for several hours. To modify with organic surfactants, this oxide form is dispersed in a clear solution of that surfactant and stirred continuously for sufficient time to allow complete regeneration of the host layer structure. In the present study, we have used this regeneration method to modify Mg–Al LDH

Table 1  
Surfactants used for intercalation into LDH

Chemical name	Structure	Functional group
Dodecylsulfate		Sulfate
Dodecyl benzenesulfonate		Sulfonate
Laurate		Carboxylate
Bis (2-ethylhexyl) hydrogenphosphate		Phosphate

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