Poster

PREPARATION AND CHARACTERIZATION OF LAYERED DOUBLE HYDROXIDES THROUGHOUT DETERMINATION OF SIZE CONTROLLABLE SYNTHETIC PARAMETERS

Sanghee Kim, Sung-Ho Hwang , Sang Kyoo Lim, Soo-Keun Lee
Advanced Nano-Materials Research Team, Division of Nano & Bio Technology, Daegu Gyeongbuk Institute of Science & Technology, 5th Floor, Daegu Technopark Venture 2 Plant, 75 Gongdanbuk2gil, Dalseo-gu, 704-230, Daegu, Republic of Korea
hsungho@dgist.ac.kr

Layered double hydroxide(LDH) compounds have attracted a great attention because of their layered structure and high anion exchange capacity, which can make various technical applications. LDHs are the only known inorganic materials with positive layer charge, in which the interlayer anions can be replaced by ion-exchange processes. Their structural units are made from stacks of positively charged octahedral sheets. The net positive charge, which is due to substitution of divalent ions with trivalent ones in the brucite-like metal hydroxide $M(OH)_2$, is balanced by an equal negative charge from the interlayer anion. The structure is also stabilized by hydrogen bonds among interlayer water molecules, anions and the hydroxyl slabs as well as by electrostatic interactions between the layer and the anions. Owing to their high anion exchange capacity, high surface area, physical strength and layered structure, LDHs have many potential applications, including pharmaceuticals, adsorbents, catalysts, catalyst supports, etc.. In recent, much attention have also been paid for the fabrication of organic-inorganic composites containing LDHs as eco-friendly materials due to their heat resistant and flame retardant properties. Especially, it is very important to how to control its particle size, which plays a critical role in such industrial applications. In this work, the synthetic parameters affecting the particle size have been investigated on the basis of crystal growth mechanism. Based on crystal growth mechanism, the total metal ion concentration, which may influence the nucleation rate, can be considered as one of the size controlling parameters. In addition, since aging time and reaction temperature could influence the crystal growth kinetically or thermodynamically, we have also considered them as the parameter to control the particle size. And two bases such as NaOH and urea were selected to determine the effects of precipitation rate on particle size, because urea shows slow hydrolysis resulting in slow precipitation. On the other hand, NaOH gives fast precipitation. Two different synthetic methods such as hydrothermal treatment and urea hydrolysis were chosen to compare the difference of basicity between strong and weak bases. In addition, we would intend to apply this technology, in more industrial point of view, to the fabrication process of the organic-inorganic composites. The nano-sized LDHs were dispersed in polyethyleneterephthalate(PET) resin, which formed master batch by using compounding technique in order to produce organic-inorganic hybrid film or fiber.

The layered double hydroxide particles of $Mg_2Al(OH)_6(CO_3)_{1/2}\cdot 0.1H_2O$ have been prepared by two different methods: (1) direct coprecipitation of aqueous solution of $Mg(NO_3)_2\cdot 6H_2O$, $Al(NO_3)_3\cdot 9H_2O$ upon hydrolysis of urea, and (2) hydrothermal aging of the same nitrate solution after NaOH titration. In order to control the particle size and morphology of LDH, various parameters such as metal ion concentration, aging time, reaction temperature are systematically studied. According to the powder X-ray diffraction, all the samples turn out to be well crystallized with an average basal spacing of 7.6 Å corresponding to the LDH crystal. From the SEM images, the coprecipitates are found to be hexagonal in shape and are controlled in the particle size range of 0.9–2.2 and 1.2–4.5 μm depending on aging time (6–69 h) and metal ion concentration (0.87–0.065 M), respectively. However, the particle size of the hydrothermally prepared samples increase in proportion to aging time (12–72 h ; 85–120 nm) and reaction temperature (100–180 °C; 115–340 nm). The effect of such parameters upon the particle size was rationalized on the basis of crystal growth mechanism.

References:

[1] F. Cavani, F. Trifiro, A. Vaccari, *Catal. Today, 11*, 173 (1991)
[2] C. O. Oriakhi, I.V. Farr, M.M. Lerner, *J. Mater. Chem., 6*, 103 (1996)
[3] J.H. Choy, S.Y. Kwak, Y. J. Jeong, J. S. Park, *Angew Chem. Int. Ed. 39*, 4041 (2000)
[4] F. Leroux, J.-P. Besse, *Chem. Mater. 13*, 3507 (2001)
[5] N.S. Puttasway, P. Vishnu Kamath, *J. Mater. Chem. 7*, 1941 (1997)

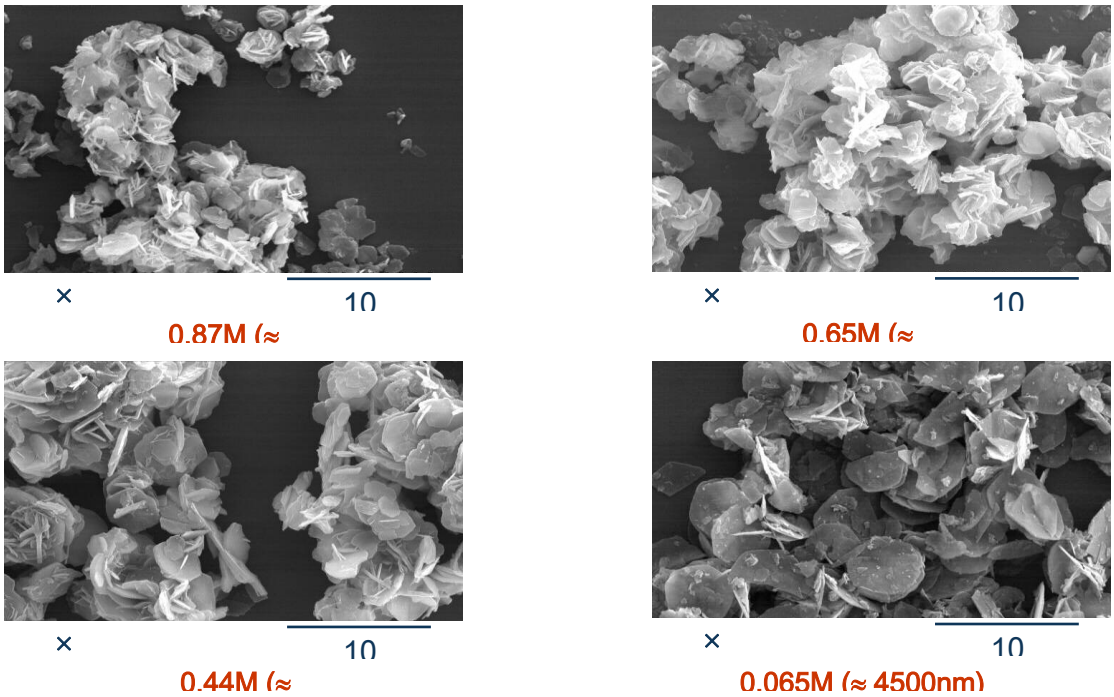

Figure 1. FE-SEM images of LDH particles prepared by urea hydrolysis.method

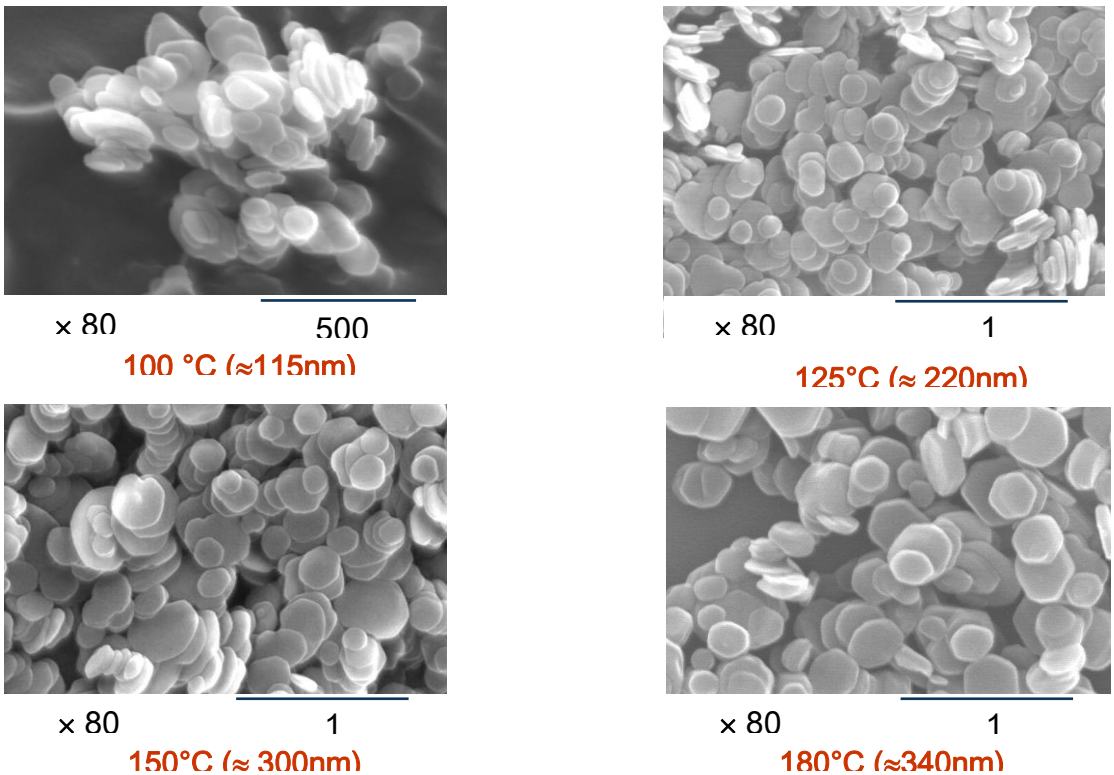

Figure 2. FE-SEM images of LDH particles prepared by hydrothermal method