

Supporting Information

Direct synthesis of hexagonal Mg(OH)₂ nanoplates from natural brucite without dissolution procedure

**Hongchang Pang, Gui-Ling Ning*, Wei-Tao Gong, Jun-Wei Ye, and
Yuan Lin**

State Key Laboratory of Fine Chemicals, School of Chemical Engineering, Dalian
University of Technology, Dalian 116024, China. E-mail: ninggl@dlut.edu.cn

Materials and Methods

In a typical synthesis, 0.025 M of brucite powder (5000 mesh) from Fengcheng in China and 0.00125 M of magnesium *p*-toluene sulfonate (TS⁻ short for *p*-toluene sulfonate, solution, pH \approx 7) were mixed in 70 mL of deionized water, then poured into a stainless Teflon-lined 100 mL capacity autoclave. The autoclave was sealed and maintained at 180 °C for 5 h, 6 h, 8 h, 12 h, 24 h, respectively, and then air cooled to room temperature. The products were washed with water several times and dried at 60 °C in air and then characterized using X-ray diffraction (XRD) for, Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM) for morphological details, high-resolution transmission electron microscopic (HRTEM) and the selected area diffraction (SAED) for crystallinity, EDX, infrared spectra (IR) and thermogravimetric analysis (TGA) for the content. In addition, to investigate the effect of organic molecules without ringed groups on morphology control, dodecyl sulfate was employed to fulfill this duty. The molar dosage of dodecyl sulfate and experimental process are exactly the same as the case of magnesium *p*-toluene sulfonate mentioned above.

Characterization

Morphology and structure of products were measured by scanning electron microscopy (SEM) and transmission electron microscope (TEM) at Electron Microscope Lab, Dalian University of Technology. The SEM (HITACHI, S-4800) and TEM (Tecnai G² 20 instrument) were operated at an accelerating voltage of 5 kV and 200 kV, respectively. The nanoparticles were dispersed in ethanol onto a film 3-4nm thick of amorphous carbon supported by 3000 mesh copper grids. Nanoparticles were deposited onto grid and ethanol was evaporated. The crystal structure and phase purity of the product were characterized by powder X-ray diffraction (XRD) on a Rigaku D/max 2400 X-ray diffractometer equipped with graphite monochromatized Cu K α radiation ($\lambda=1.5406$ Å). A scan rate of 0.02 ° s⁻¹ was applied to record the pattern in the 2 θ range from 10 ° to 80 °. Infrared spectra (IR) were recorded on a JASCO FT/IR-460 plus spectrometer using KBr technique. Thermogravimetric (TG) analysis was performed on TGA/SDTA 851^e thermal analyzer at a heating rate of 10 °C/min under a static atmosphere.

Fig. S1 EDX of the nanoplates magnesium hydroxide synthesized reveals that no S was detected. It is proved that magnesium *p*-toluene sulfonate has not been consumed. In other words, the magnesium *p*-toluene sulfonate solution could be recycled to use as mother solution next time. Further evidence would be seen in IR spectra (Fig. S7) and TG analysis (Fig. S8).

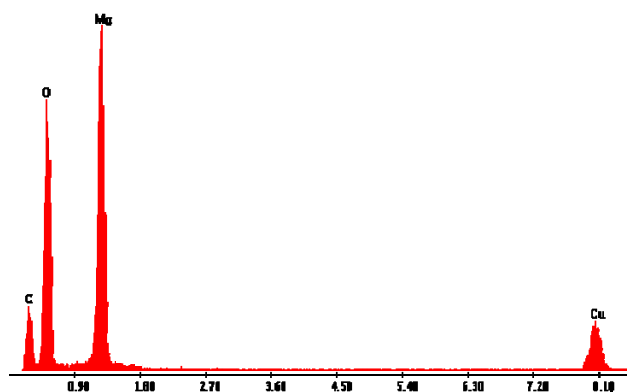


Fig. S2 TEM image of as-synthesized $\text{Mg}(\text{OH})_2$ by using 10 times cyclic magnesium *p*-toluene sulfonate mother solution.

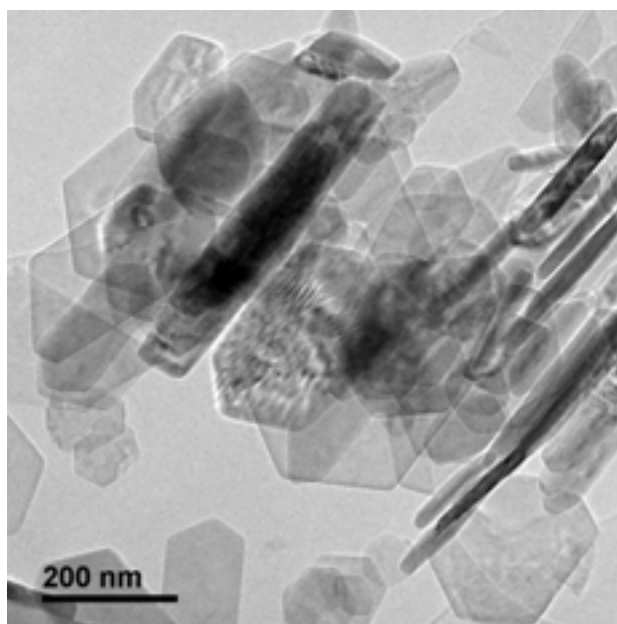


Fig. S3 (a) SEM image of natural brucite surface. Layered structure would be ready for disaggregation into thin nanosheets. (b) Structural illustration of natural brucite.

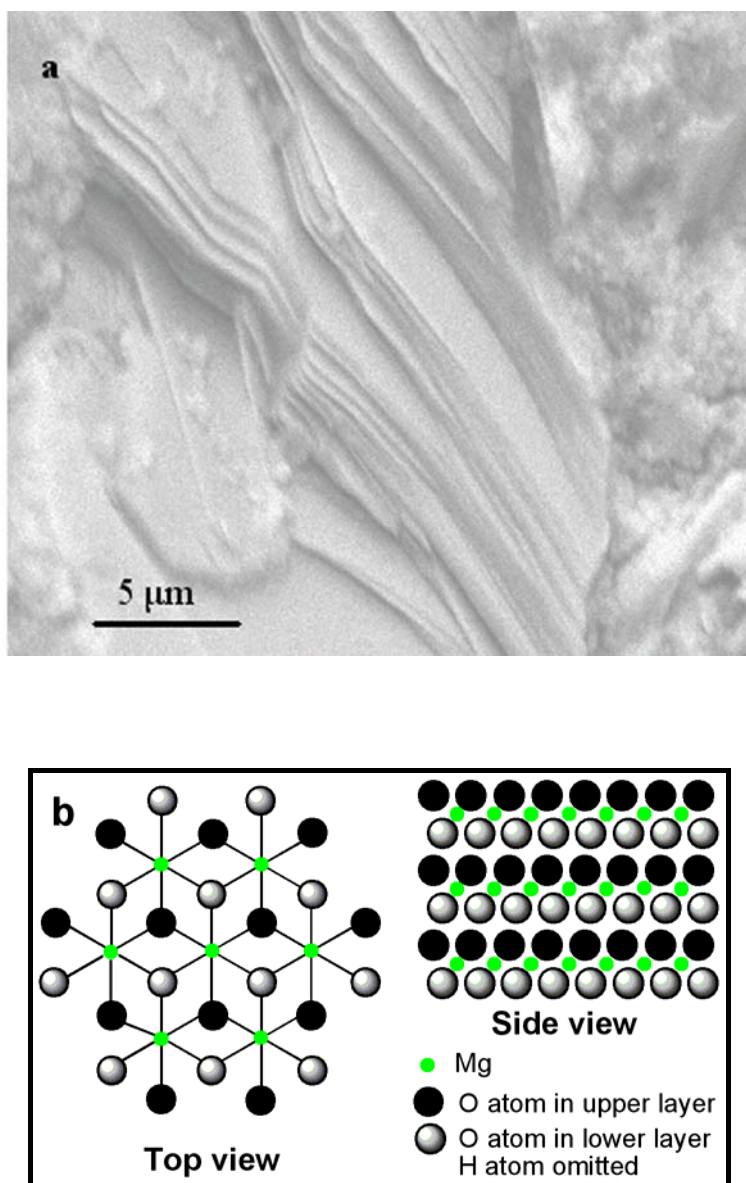


Fig. S4 XRD patterns of (a) (b) and (c) intermediate products reacted for 5 h, 8 h and 12 h, respectively, demonstrated the gradual evolution of natural brucite from original mineral phase (a), via metastable phase (b), to synthetic $\text{Mg}(\text{OH})_2$ phase (c).

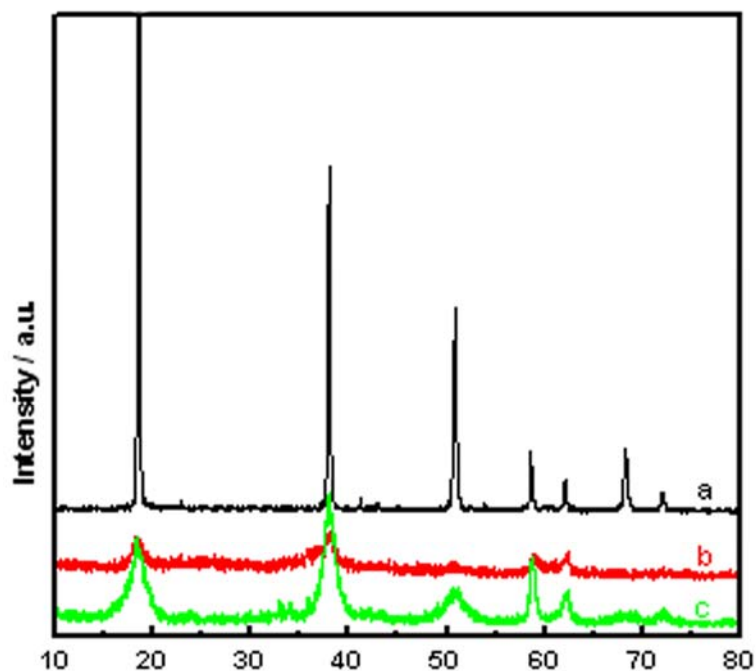


Fig. S5 (a) XRD pattern of raw material brucite. (b) XRD pattern of the product synthesized at ambient temperature and pressure in the presence of TS⁻.

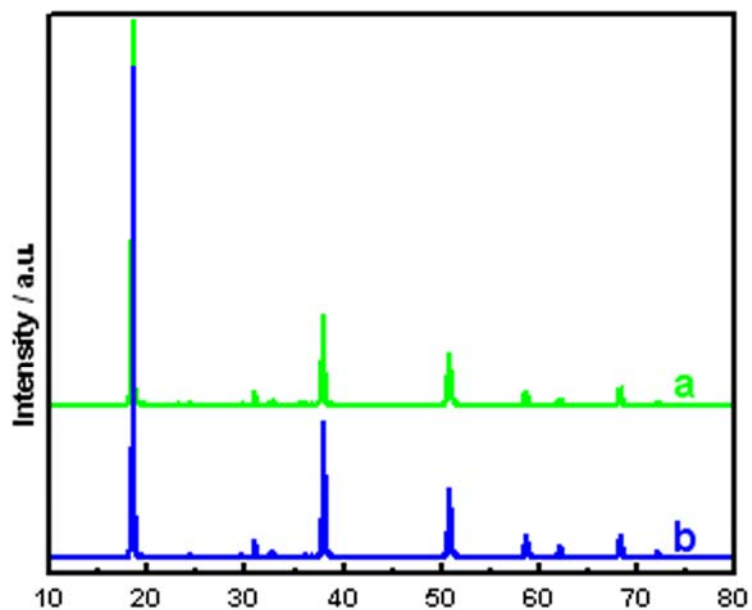


Fig. S6 TEM image of the product synthesized at room temperature and normal atmosphere in the presence of TS^- indicated that brucite did not go through disaggregation and remained the lattice feature of raw material brucite.

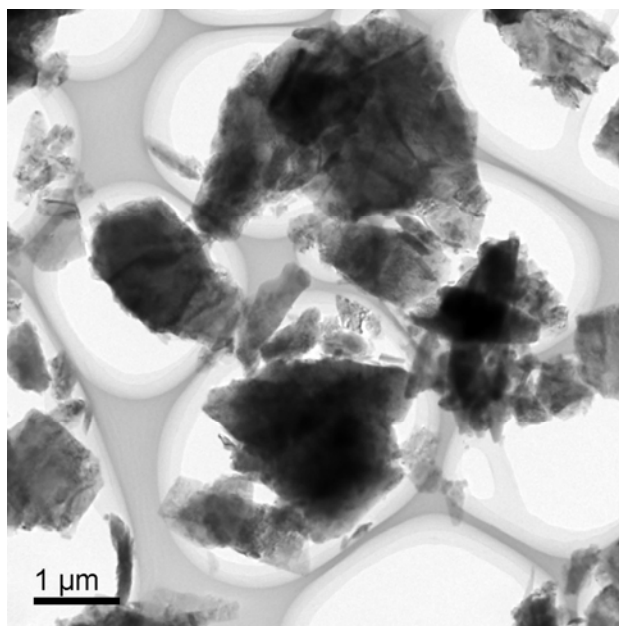


Fig. S7 IR spectra of the as-synthesized hexagonal $\text{Mg}(\text{OH})_2$ nanoplates and natural brucite. In comparison with raw material brucite, no absorbed bands of organic groups were detected in the final product. It also indicated that TS^- organic groups have not been bonded in the as-synthesized $\text{Mg}(\text{OH})_2$ product.

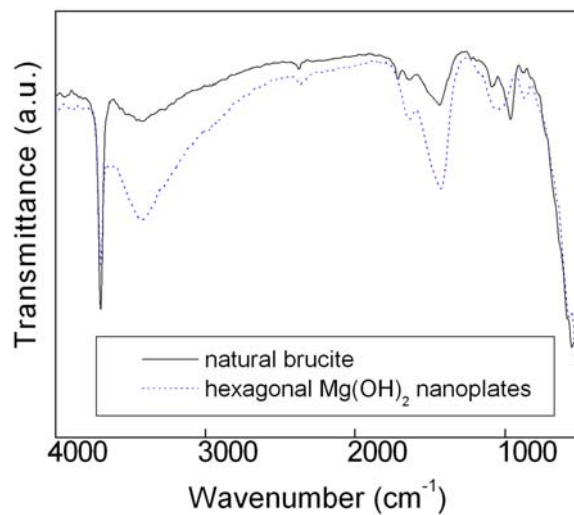


Fig. S8 TG/TGA curve of the as-synthesized hexagonal $\text{Mg}(\text{OH})_2$ nanoplates. The TG curve shows that the obtained $\text{Mg}(\text{OH})_2$ nanoplates was decomposed through a pronounced weight loss process from 330 to 430 $^\circ\text{C}$, and a corresponding derivative weight peak is observed near 393 $^\circ\text{C}$. This can be clearly attributed to the decomposition of $\text{Mg}(\text{OH})_2$. However, the theoretical weight loss of $\text{Mg}(\text{OH})_2$ transforming to MgO is 30.8%, which is quite accordant with the observed 30.5%. These data suggest that TS^- organic group would not be incorporated into the as-synthesized $\text{Mg}(\text{OH})_2$ product.

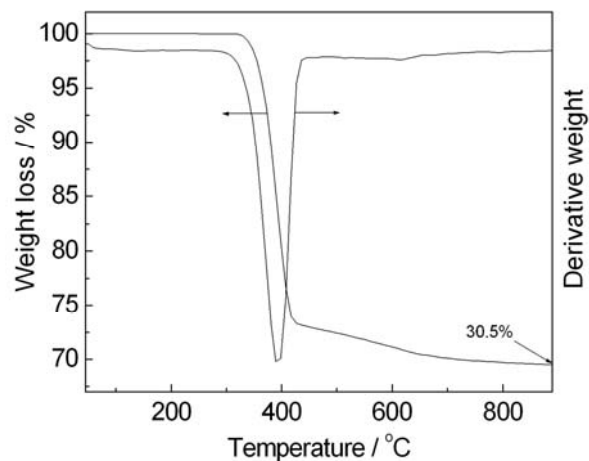


Fig. S9 Typical TEM image of intermediate product reacted for 6 h. Nanosheets originated from disaggregation of natural brucite and primary nucleus have been observed, which strongly verified the transformation process.

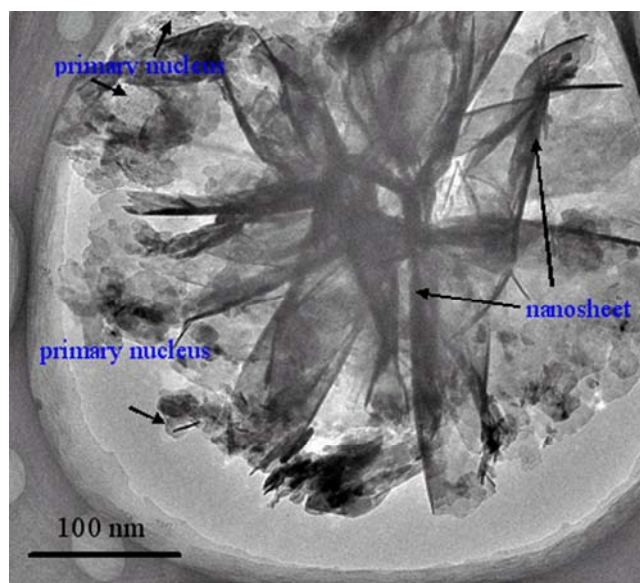
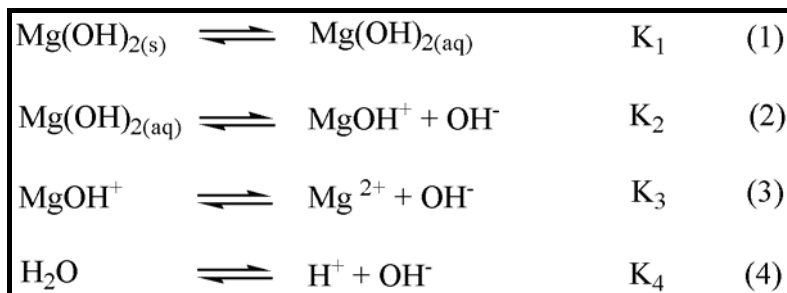


Table S1 Equilibrium constants for reaction (1) ~ (4) at different temperatures^{1,2}, suggest that natural brucite would be still insoluble even at high 220 °C. In other words, direct synthesis from bulk brucite particles to Mg(OH)₂ nanoplates through hydrothermal process at 180 °C should exclude dissolution-recrystallization procedure.



T/°C	K ₁	K ₂	K ₃	K ₄
140	2.88 x 10 ⁻¹²	5.71 x 10 ⁻²	4.93	1.77 x 10 ⁻¹²
160	2.43 x 10 ⁻¹²	1.33 x 10 ⁻¹	1.44	2.66 x 10 ⁻¹²
180	2.03 x 10 ⁻¹²	2.82 x 10 ⁻¹	4.43 x 10 ⁻¹	3.66 x 10 ⁻¹²
200	1.685x 10 ⁻¹²	5.52 x 10 ⁻¹	1.44 x 10 ⁻¹	4.64 x 10 ⁻¹²
220	1.39x 10 ⁻¹²	1.01	4.87 x 10 ⁻²	5.48 x 10 ⁻¹²

References:

- 1 Outokumpu Research Oy HSC Chemistry, Finland, 1993.
- 2 L. Xiang, Y. C. Jin, Y. Jin, *Chinese Journal of Inorganic Chemistry*, 2003, **19**, 837.