FOUNDATIONS
ADVANCES

Volume 72 (2016)

Supporting information for article:

Thermal evolution of Mg–Al and Ni–Al layered double hydroxides: the structure of the dehydrated phase

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Thermal gravimetric analysis

On the DTG curves of $Mg_3Al\text{-}CO_3$, $Mg_2Al\text{-}CO_3$ and Mg_2Al-OH LDH samples (Figure S1), three regions of weight loss are observed over the temperature range of 300–1300 K. In the first region, at temperatures below 423 K, the removal of physisorbed water occurs; in the second region, at 423–573 K, interlayer water is removed; and in the third region, at 573–873 K, which is represented by two overlapping peaks, dehydroxylation of the layers and removal of carbonate anions take place.

For $Ni_3Al\text{-}CO_3$ LDH, the first and second weight losses at temperatures below 423 K and in the range of 423–523 K on the DTG curve are also related to the removal of physisorbed water and dehydration – removal of water molecules from interlayer spaces. The third weight loss region at 523–723 K is represented by a single broad peak that is caused by simultaneous dehydroxylation of the brucite layers and the removal of carbonate anions from interlayer spaces.

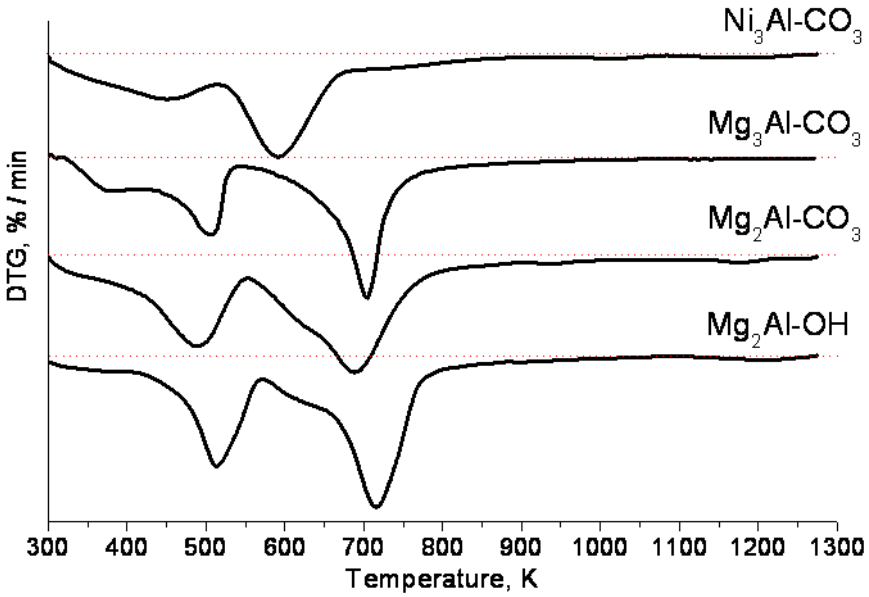
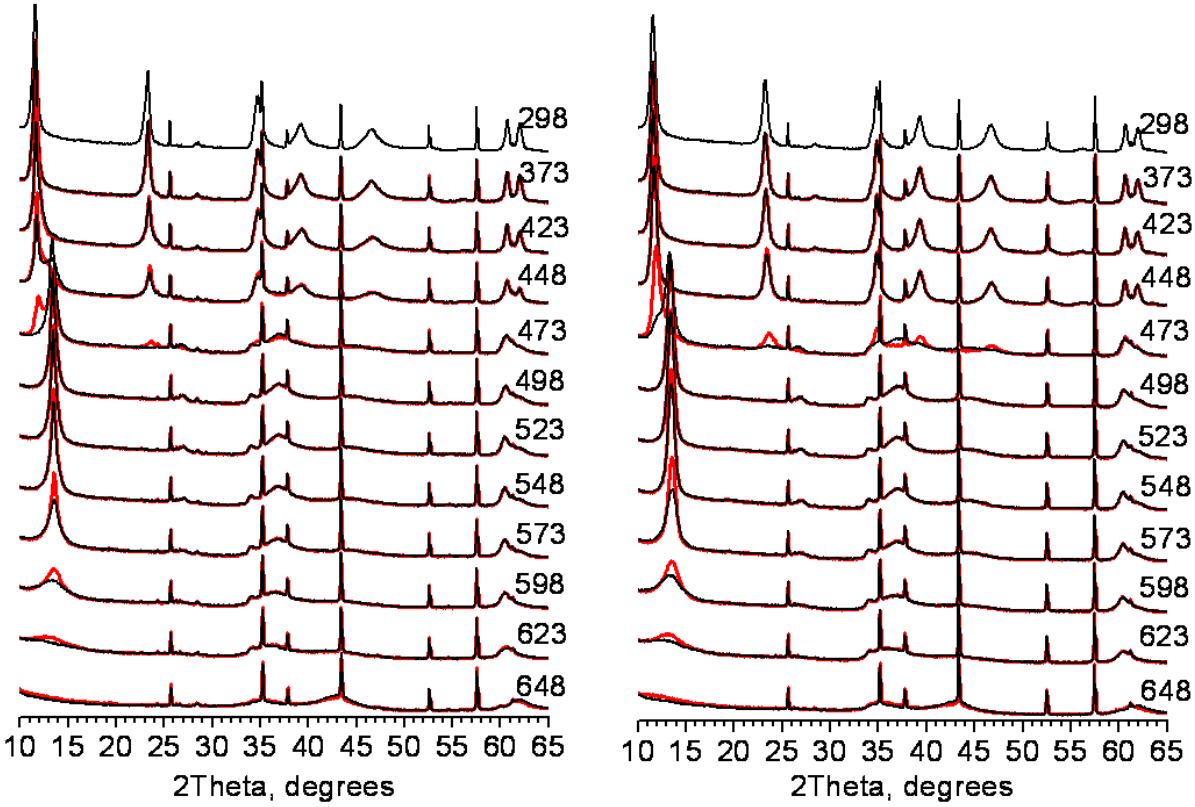


Figure S1 DTG curves.



(*a*) (*b*)

Figure S2 XRD patterns recorded from room temperature to 648 K for the samples: (*a*) $Mg_2Al\text{-}CO_3$; and (*b*) $Mg_2Al\text{-}OH$.

Table S1 Angular shifts of the peaks in dependence on polytype ratio $3R_1$:1H:$3R_2$ in the dehydrated phase for the Mg_3Al-CO_3 sample ($3R_1$=1-1H-$3R_2$).

$3R_2$	1H,0.0	1H,0.1	1H,0.2	1H,0.3	1H,0.4	1H,0.5	1H,0.6	1H,0.7	1H,0.8	1H,0.9	1H,1.0
0.0	1.53 3.71	1.51 3.66	1.46 3.57	1.40 3.38	1.33 2.88	1.10 2.32	0.88 1.87	0.69 1.53	0.59 1.30	0.4 / 1.13	0.32 / 0.98
0.1	1.61 3.98	1.59 3.83	1.56 3.67	1.51 3.35	1.30 3.19	0.93 2.20	0.77 1.57	0.51 1.25	0.35 0.99	0.25 / 0.83	
0.2	1.65 4.11	1.63 4.04	1.57 3.85	1.44 3.59	1.18 2.61	0.81 1.53	0.39 1.08	0.24 0.78	0.13 0.67		
0.3	1.69 4.37	1.64 4.32	1.59 4.27	1.50 3.71	0.80 1.37	0.23 0.52	0.05 0.43	0.0 0.38			
0.4	-3.24 -4.22	-3.15 -3.94	-2.82 -3.29	-1.51 -2.01	-1.23 -0.28	-0.24 0.0	-0.24 0.05				
0.5	-2.82 -3.70	-2.40 -3.24	-1.74 -2.30	-1.03 -1.54	-0.61 -0.89	-0.47 -0.51					
0.6	-2.35 -3.24	-2.02 -2.53	-1.55 -2.16	-1.08 -1.41	-0.8 -0.93						
0.7	-2.11 -2.81	-1.79 -2.30	-1.46 -1.92	-1.13 -1.36							
0.8	-1.88 / -2.48	-1.65 / -2.16	-1.37 / -1.73								
0.9	-1.73 / -2.21	-1.51 / -1.83									
1.0	-1.62 / -2.09										

Table S2 Angular shifts of the peaks in dependence on polytype ratio $3R_1$:1H:$3R_2$ in the dehydrated phase for the Ni_3Al-CO_3 sample ($3R_1$=1-1H-$3R_2$).

$3R_2$	1H,0.0	1H,0.1	1H,0.2	1H,0.3	1H,0.4	1H,0.5	1H,0.6	1H,0.7	1H,0.8	1H,0.9	1H,1.0
0.0	-0.87 -3.77	-0.91 -3.88	-0.98 -3.97	0.07 2.1	0.12 2.05	0.14 1.81	0.17 1.48	0.19 1.1	0.23 0.97	0.24 0.89	0.26 0.80
0.1	-0.87 -3.71	-1.01 -3.83	-0.98 -3.94	0.04 2.12	0.07 2.03	0.11 1.64	0.14 1.28	0.18 0.99	0.20 0.86	0.22 0.78	
0.2	-0.81 -3.49	-0.9 -3.66	-0.98 -3.83	0.03 2.15	0.08 1.89	0.09 1.49	0.12 1.0	0.15 0.84	0.19 0.72		
0.3	-0.75 -3.38	-0.74 -3.55	-0.70 -3.71	-0.61 2.2	-0.07 1.84	0.02 1.05	0.08 0.8	0.15 0.6			
0.4	-0.68 -3.15	-0.63 -2.99	-0.62 -2.78	-0.15 2.23	0.0 1.23	0.06 0.54	0.1 0.49				
0.5	-0.53 -2.3	-0.42 -2.08	-0.25 -1.51	-0.02 -0.75	0.0 -0.13	0.09 0.14					
0.6	-0.41 -2.05	-0.28 -1.62	-0.14 -1.2	-0.06 -0.7	0.0 -0.36						
0.7	-0.30 -1.8	-0.22 -1.41	-0.11 -1.01	-0.05 -0.65							
0.8	-0.22 -1.57	-0.14 -1.23	-0.09 -0.9								
0.9	-0.17 -1.41	-0.09 -1.16									
1.0	-0.14 -1.29										