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Insight into Structural Change of Ni(II)-Al(III) Layered Double Hydroxide under Heat Treatment: Design of NiO-Al$_2$O$_3$ Nanocomposite Catalysts toward Hydrogen Transfer Reaction of Furfural

In this study, a Ni$_2$AlO_T catalyst derived from the calcination at T K of CO$_3^{2-}$/Ni$_2$Al LDH, obtained by the hydrothermal method using urea,[1] was synthesized. To clarify the structural change of CO$_3^{2-}$/Ni$_2$Al LDH during calcination, temperature-programmed synchrotron XRD, TG-DTA, and Ni K-edge XANES (Fig. 1) were obtained under the same heating conditions (rate: 10 K/min). XRD data revealed the Ni$_2$AlO_T catalyst (T $\geq$ 673 K) was mainly composed of small sized NiO species and amorphous Al$_2$O$_3$. In addition, the decomposition of the hydroxide nanosheets into NiO species around 653 K was also confirmed. The Ni$_2$AlO_T catalysts were applied to hydrogen transfer reaction of furfural with 2-propanol (Table 1). The yield of furfuryl alcohol increased with increasing calcination temperature. From the H$_2$-TPR measurements, it was found that a highly stable NiO species against the reduction into Ni(0) was generated on the Ni$_2$AlO_T matrix by the calcination at high temperature.
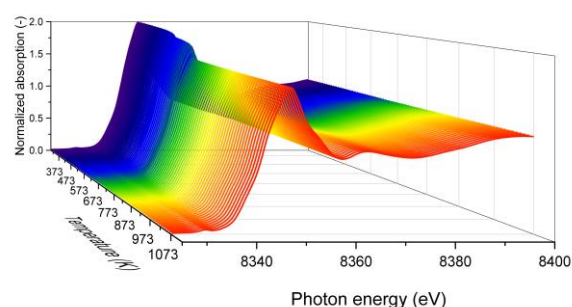
Fig. 1 Normalized Ni K-edge XANES for the CO$_3^{2-}$/Ni$_2$Al LDH as a function of temperature programmed calcination (10 K/min) under air flow

Table 1. Results of hydrogen transfer reaction of furfural with 2-propanol[a]

Entry	Catalyst	Conv. (%)[b]	Yield (%)[b]
1	Blank	-	n.d.
2	NiO	10	n.d.
3	Ni$_2$AlO_673	35	35
4	Ni$_2$AlO_773	32	32
5	Ni$_2$AlO_873	44	44
6	Ni$_2$AlO_973	43	43
7	Ni$_2$AlO_1073	45	45
8	Ni$_2$AlO_1173	51	51

[a]Catalyst (0.1 g), furfural (1 mmol), 2-propanol (5 mL), 373 K, 3 h. [b]Determined by GC using internal standard technique.

1) R. Sasai, *et al.*, *Bull. Chem. Soc. Jpn.*, 95, 802-812 (**2022**).