

EFFECT OF THE Ni-Al HYDROTALCITE-LIKE PRECURSOR HYDROTHERMAL TREATMENT ON THE PROPERTIES OF THE RELATED MIXED OXIDE CATALYST

KOVANDA, F.,¹ ROJKA, T.,¹ JIRÁTOVÁ, K.,² OBALOVÁ, L.³

¹ Department of Solid State Chemistry, Institute of Chemical Technology [Ústav chemie pevných látek, Vysoká škola chemicko-technologická], Technická 5, Praha, 166 28, Czech Republic

² Institute of Chemical Process Fundamentals [Ústav teoretických základů chemické techniky], Rozvojová 135, Praha, 165 02, Czech Republic

³ Department of Physical Chemistry and Theory of Technological Processes, Technical University of Ostrava [Katedra fyzikální chemie a teorie technologických procesů, Technická univerzita Ostrava], tř. 17. listopadu 15, Ostrava–Poruba, 708 33, Czech Republic

E-mail: Frantisek.Kovanda@vscht.cz

The well dispersed homogeneously mixed oxides are formed during thermal decomposition of hydrotalcite-like compounds (layered double hydroxides) and therefore the hydrotalcite-like precursors are often used for preparation of mixed oxide based catalysts. Hydrothermal treatment of the precursors may increase their crystallinity and particle size, depending mainly on temperature and time of the crystallisation process. The layered crystal structure of hydrotalcite-like compounds collapses during the thermal decomposition and the oxides obtained at moderate temperatures are rather amorphous but a relation between the precursor crystallinity and properties of prepared catalysts may be considered.

The Ni-Al layered double hydroxide with Ni/Al molar ratio of 2 was prepared by coprecipitation. The obtained suspension was hydrothermally treated in autoclaves under autogeneous water vapour pressure at 180°C for 4–20 hours. The washed and dried precursors were then formed into extrudates and calcined at 450°C. The prepared precursors and calcined products were characterised by powder X-ray diffraction, scanning electron microscopy, BET surface area and pore size measurements, Raman spectroscopy and temperature programmed reduction. The prepared Ni-Al mixed oxides were also tested in the catalytic decomposition of N₂O.

A hydrotalcite-like phase of relatively low crystallinity was present in the coprecipitated product. Together with hydrotalcite diffraction lines, some slight unidentified diffraction lines were found in the powder XRD patterns of hydrothermally treated samples. The integral intensity and full width in half maximum (FWHM) of diffraction (003) and (006) lines were evaluated to compare the crystallinity of samples hydrothermally treated for various times. The integral intensity of both diffraction lines increased with time of hydro-

thermal treatment. A significant decrease of FWHM values in a relatively short time (4 hours) was observed, then the time dependence of the FWHM values was less evident. The hydrothermal treatment caused also a decrease in the surface area and porosity of the coprecipitated product. The SEM images showed a considerable increase of crystal size of the hydrothermally treated samples.

Samples calcined at 450°C contained NiO (bunsenite), no other crystalline phases were detected. The XRD patterns of calcined samples showed only a slight difference in the integral intensity of NiO diffraction lines but a gradual decrease of the FWHM values with increasing time of the precursor hydrothermal treatment was observed. The hydrothermal crystallisation of precursors considerably influenced the reducibility of calcined samples. The maximum reduction of the studied mixed oxides shifted to higher temperatures with the time of precursor hydrothermal treatment (from 525°C measured for sample without hydrothermal treatment to 580°C for sample treated for 20 hours). The total H₂ consumption in the temperature interval 20–800°C was nearly the same for all calcined samples but the H₂ consumption measured between 350 and 450°C decreased with time of hydrothermal treatment. On the other hand, the hydrothermal treatment of precursors slightly increased the catalytic activity of obtained mixed oxides in the catalytic N₂O decomposition.

Acknowledgements

This work was supported by the Czech Ministry of Education, Youth and Sports (research project no. CEZ:MSM 223-10-0002) and by the Grant Agency of Czech Republic (projects no. 106-02-0523 and 104-04-2116).

Table 1:

Time of precursor hydrothermal treatment [hour]	FWHM of diffraction lines [2θ]		H ₂ consumption (350–450°C) [mmol H ₂ /g]	T ₅₀ * [°C]
	hydrotalcite (003)	NiO (200)		
0	4.083	3.820	0.72	382
4	0.591	3.217	0.29	374
8	0.509	2.679	0.24	378
20	0.393	2.460	0.17	370

* Temperature at which 50% conversion of N₂O was achieved (0.1 g of catalyst, 1000 ppm N₂O in He, flow rate 100 ml min⁻¹)