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Synthesis of Cu-based ex-hydrotalcite catalysts - a structural characterization and a first look to influencing parameters

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Introduction

Cu,Zn,Al hydrotalcite-like compounds (or layered double hydroxides, LDH) are formed as a by-phase during the synthesis of the precursors of the well known Cu/ZnO/Al₂O₃ catalysts for Methanol synthesis. As phasepure Cu,Zn,Al hydrotalcites, which have the general composition $(\text{Cu}_{1-x}\text{Zn}_x)_{1-y}\text{Al}_y(\text{OH})_2(\text{CO}_3)_{y/2} \cdot 2\text{H}_2\text{O}$ [1], have a perfect distribution of all three metal components and a homogeneous microstructure they are interesting for deeper investigations.

Such LDH's can be obtained in a phase pure, Cu-rich form by co-precipitation with NaOH/Na₂CO₃, which means that both the metal salt solution and the base are dosed simultaneously using a constant pH method at 25 °C. Upon calcination in air the precursor was converted into the mixed oxides. Finally, the Cu-based catalyst was obtained by reduction of the calcined sample in 5% H₂/Helium.

In our contribution we will present the structural characterization (XRD, BET, IR, SEM, TG-MS, TPR and TEM) of this catalyst and its precursors as well as the investigation of some influencing synthesis parameters like pH.

Results and discussion

The products of the co-precipitation have a hydrotalcite-like structure (Fig. 1a). No other crystalline phases can be detected. As expected the particles appear in a platelet-like morphology which is maintained during calcination (Fig. 1b).

By SEM investigations (Table1) the biggest particle size was found for pH 7 and pH 10 and the smallest one for pH 8. These results are in a quite good agreement with the surface areas determined with the BET method (see Fig. 1a). The most homogeneous particle size distribution was obtained at pH 10 and the most inhomogeneous one at pH 7 which was also measured by SEM.

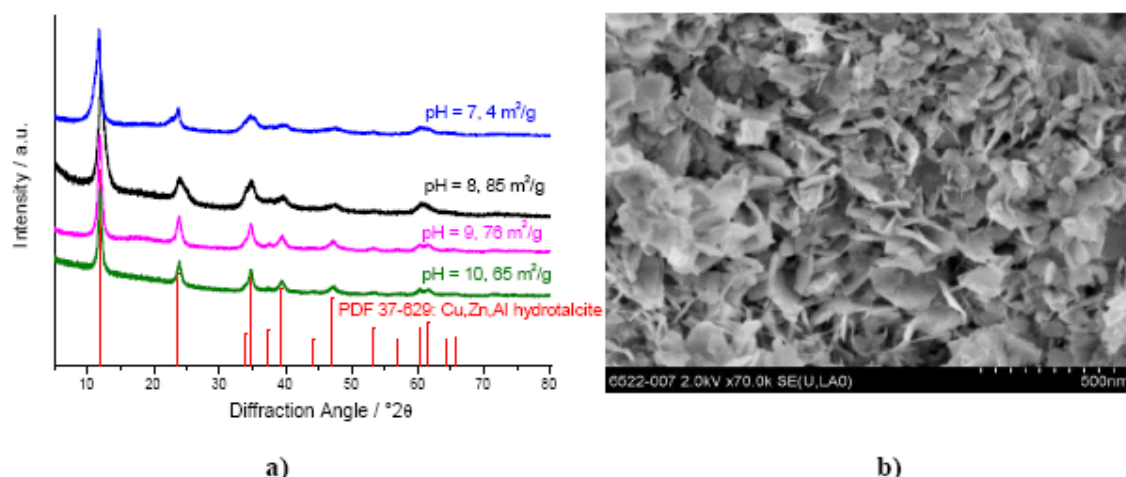


Figure 1: a) XRD pattern of htl precursor materials in dependence of the synthesis pH value; b) SEM image of the sample after calcination

Table 1: particle size and particle size distribution of the htl precursors obtained at different pH values measured by SEM (respective 50 particles)

pH	mean	min	max	max/min
7	104,16 nm	50,26 nm	161,93 nm	3.22
8	86,63 nm	48,29 nm	134,00 nm	2.77
9	96,55 nm	62,50 nm	171,52 nm	2.74
10	113,44 nm	74,08 nm	187,28 nm	2.53

Conclusions

Phasepure Cu,Zn,Al hydrotalcite was obtained with a high Cu content by a co-precipitation route. The best synthesis pH value was pH 8 yielding Cu based catalysts with a high surface area and small Cu particles.

Acknowledgements

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References

- [1] F. Cavani, F. Trifirò, A. Vaccari, *Catal. Today* **11** (1991) 173.