

Synthesis of Mesoporous Zeolite LaX by Sequential Steaming and Acid Leaching

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Abstract

Zeolites are a widely used class of microporous solid acids utilized for catalysis, and especially large pore zeolites are often applied in industrial refining processes.^[1] Mainly in the past few years, synthesis of hierarchically zeolites, which provide mesopores besides micropores, came into research focus.^[2] By usage of hierarchical zeolites, diffusion limitation problems may be overcome by improvement of mass transport.^[1-2] A very common synthesis of mesoporous zeolite is the dealumination of NH₄Y by steam treatment, resulting in a more hydrothermal stable USY. Since zeolite X has a very high aluminum content, dealumination by steam-treatment results in the collapse of the zeolite structure, since only an insufficient amount of Si-atoms is present to refill vacancies.^[3] By ion-exchange of NaX with La³⁺ ions, the zeolite structure is stabilized and thus zeolite LaX is less prone to water-vapor.^[1]

LaX was used as the starting material for steam treatment. ²⁷Al MAS NMR showed the formation of extra-framework aluminium after steam treatment (Figure 1). Since vapor treatment does not lead to formation of mesopores itself, mild acid washing was applied to remove aluminum debris. Various acids were tested, e.g. HCl, oxalic acid, tartaric acid or Na₂H₂EDTA. Analysis of the N₂ isotherms revealed the formation of various amounts of mesopores. For oxalic treated sample, this is also seen in the SEM picture, which shows a roughening of the zeolite surface after treatment (Figure 2). However, a disadvantage of some acids, for example tartaric acid, is the simultaneous destruction of micropores.

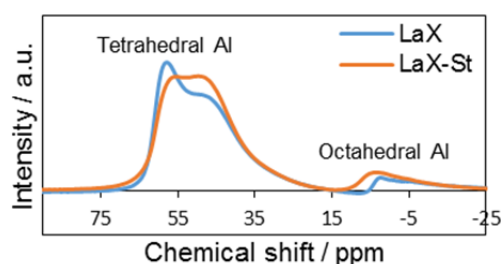


Figure 1: ²⁷Al NMR of LaX and steamed LaX.

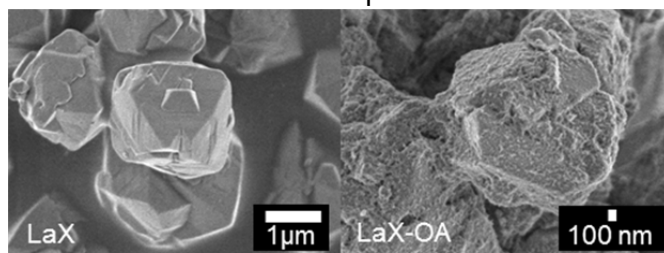


Figure 2: SEM pictures of LaX and oxalic treated LaX.

References:

- [1] F. Schüßler, *Impact of La³⁺ cations in FAU zeolites to hydride transfer reactions*, Technical University of Munich (Munich), **2013**.
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