Adsorption of acetone on silica-alumina gels

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Motivation

The recovery of the greenhouse gas CO_2 from industrial processes via carbon capture technologies is of increasing importance. The captured CO_2 must be conditioned for storage or further utilization. Among other steps, drying is carried out using adsorption on oxide-based adsorbents. During this process, accompanying compounds such as ketones and aldehydes are co-adsorbed. Under repeated adsorption–desorption cycling with a typical temperature range in industrial processes of 25 °C (adsorption) and 175 °C (desorption), these compounds polymerize on the adsorbent surface. These conditions lead to material aging and a reduction of adsorption capacity. Understanding these mechanisms is of great interest to reduce aging effects. Therefore, adsorption and desorption experiments with acetone on silica–alumina gels are performed. Temperature-programmed desorption is complemented by thermogravimetric analysis up to 800 °C. The influence of the adsorption temperature is investigated to obtain a deeper understanding of the formation of reaction products.

Methods and Materials

Methods:

1. Adsorption of acetone on silica alumina gel
2. Concentration Swing Desorption with pure N_2 at adsorption temperature
3. Temperature Programmed Desorption with pure N_2 up to 175 °C
4. Thermogravimetric Analysis with pure N_2 up to 800 °C

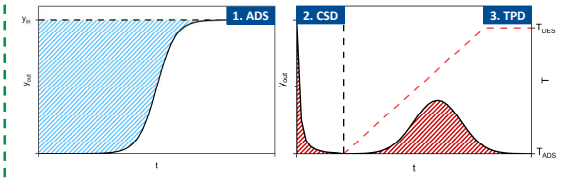

Fig. 4: Breakthrough curves of Adsorption and Desorption

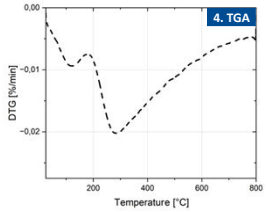

Fig. 5: Mass change curve of thermogravimetric analysis

Experimental setup:

Adsorption and Desorption:

- T: 25 °C to 175 °C
- p: 1.3 bar

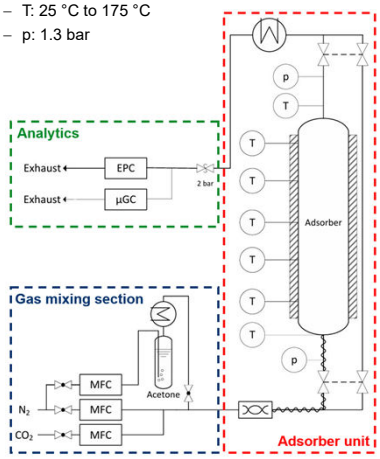

Fig. 1: Experimental setup

Silica-Alumina:

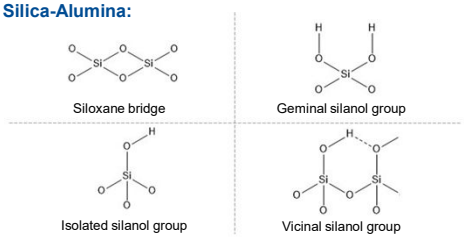

Fig. 2: Functional groups on silica surface

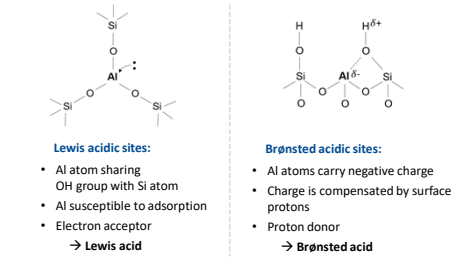

Fig. 3: Lewis- and Brønsted acid sites on silica-alumina surface

Results and Discussion

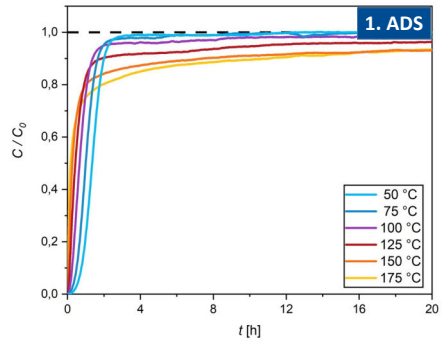

Fig. 6: Breakthrough curves of Adsorption at 25 – 175 °C

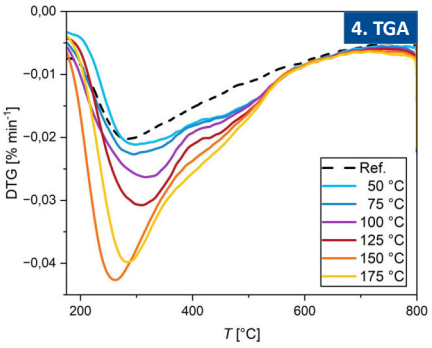

Fig. 7: Mass change curve of TGA after TPD

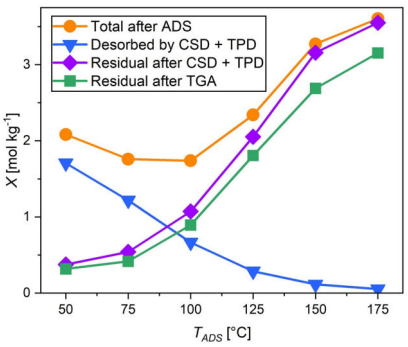

Fig. 8: Calculated Loadings

Temperature effects during adsorption:

Mass transport kinetics:

- Faster kinetics with increasing temperature
- ➢ Steeper initial slope of the BTC at high temperatures

Capacity:

- Physisorptive capacity decreases with increasing temperature
- ➢ Area above the curve should decrease at high temperatures

Reaction kinetics:

- Faster reaction kinetics with increasing temperature
- ➢ Reaction becomes more favored at high temperatures, leading to a slow approach to the outlet concentration and high loading

Investigation of reaction products:

Reference measurement:

- Unloaded Sorbead R, pre-treated under same conditions as for the adsorption experiments
- Mass loss indicates dehydroxylation of surface OH-groups

TGA curves after TPD:

- 2 peaks at 300 and 450 °C
- ➢ 2 different binding mechanisms or reaction products
- Increasing trend up to 150 °C
- At 175 °C smaller mass change despite higher residual loading
- ➢ Another binding mechanism must occur

Quantitative evaluation:

- Total desorbed amount decreases with rising temperature due to decreasing physisorptive capacity
- Residual loading after TPD increases with temperature, indicating higher reactivity
- Significant residual loading after TGA:

➢ **Reaction leads to structures thermally stable up to 800 °C**

➢ **High process temperatures seem to be crucial for aging effects**

Summary and Outlook

The breakthrough curves revealed a decrease in physisorptive capacity with increasing temperature, while at high adsorption temperatures chemical reactions dominate the process. The calculated loadings underline this trend, showing a strong increase in residual loading after desorption as the adsorption temperature rises. During TGA, the chemisorbed species were only partially desorbed, resulting in a significant residual loading even after heating up to 800 °C. This indicates that thermally stable surface structures are formed, which are responsible for material aging effects. In future work, the chemical nature of these surface structures should be analyzed in greater detail. Further investigations into the influence of process conditions will be carried out to obtain a deeper understanding of the underlying mechanisms and to identify strategies to mitigate aging effects.

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