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Alumina-based desiccants modified with potassium, rubidium, and cesium cations: surface properties and kinetics of adsorption

Eugene P. Meshcheryakov, Mariya P. Shcherbakova-Sandu, Sergey I. Reshetnikov and Irina A. Kurzina

Materials and methods

All reagents used to modify the alumina were of analytical grade. A caesium hydroxide (CsOH) solution, rubidium hydroxide hydrate ($RuOH \cdot H_2O$), and potassium hydroxide (KOH) were all obtained from Sigma–Aldrich.

The method of impregnation from excess solution was used to modify aluminum oxide granules. 300 ml of distilled water was poured into a litre glass beaker; the calculated amount of the corresponding alkali (KOH, RbOH or CsOH) was added and stirred until it was completely dissolved. Then, adsorbent granules (200 g) were immersed in this solution and kept for 24 hours while stirring, where after they were filtered and dried at 120°C. Subsequently, the samples were calcined at 500°C for 4 hours in an electric muffle furnace.

The phase composition of the samples was determined using a Rigaku Miniflex 600 X-ray diffractometer with Cu-Kα radiation at 40 kV and 15 mA, whose scanning range was $10° < 2\theta < 90°$ and the shooting speed was 2 deg/min. The acquired data were decoded using the International Centre for Diffraction Data (ICDD) database, version PDF2.

The texture of the adsorbents was determined using nitrogen adsorption isotherms at 77 K with a 3Flex automatic gas adsorption analyser (Micromeritics, USA). The specific surface area was measured using the BET method. The pore size and pore volume were determined using the Barrett-Joyner-Halenda (BJH) method.

The mass fraction of the alkali metals in the samples was determined using an XRF-1800 Sequential X-ray Fluorescence Spectrometer (Shimazu, Japan).

An ITAN pH meter/ionomer (Russia) was used to measure the acid-base characteristics of the samples. 40 ml of double-distilled water with pH = 6.4-6.7 was poured into a 50 ml glass beaker. Using the magnetic stirrer, we stirred the water until the potential of the glass electrode stabilised. Then, we added the sample (0.4 g) to the beaker and began measuring by starting the stopwatch. To characterise the acid-base state of the surface, two parameters were chosen: pH after 15 s of contact of the sample with water (pH_{15}) and the pH isoionic point (pH_{iip}), which characterises the equilibrium state.

The kinetics of water vapour adsorption was studied with a laboratory device with a McBain-Bakr quartz spring balance using samples with a particle size of 0.5-1 mm. The sensitivity of the balance was $2.9\text{-}10^{-3}$ g/mm. A V-630 cathetometer was used to measure the elongation of the spiral during adsorption. Before the adsorption studies, each sample was regenerated in a flow of high-purity Ar (5 L/h) at 200°C for one hour. The experiments were conducted at 25 °C. For the water vapour adsorption in 100% humidity, argon was supplied to the sample at a gas flow rate of 30 L/h, passed through two Dreschel bottles filled with distilled water.

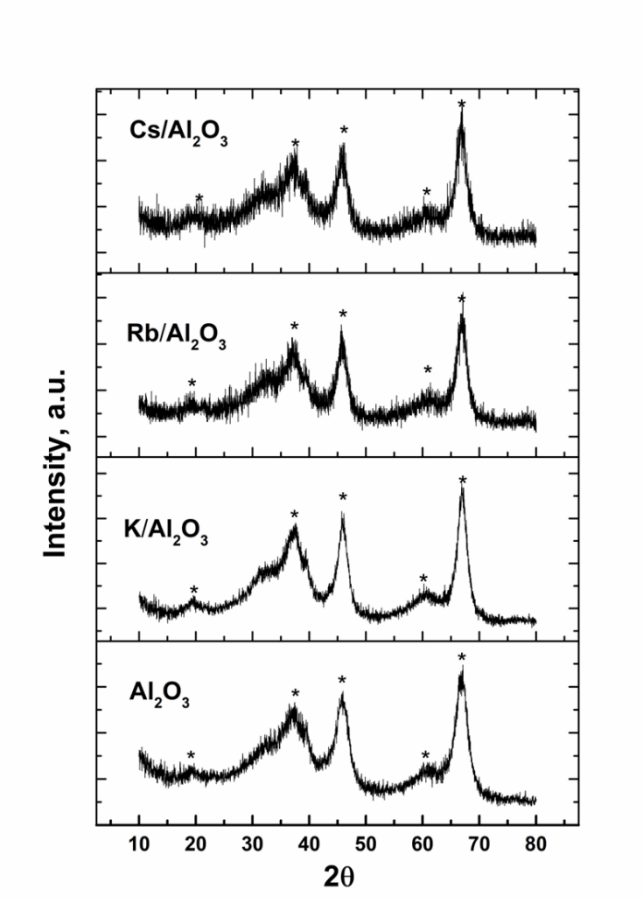

Figure S1 XRD analysis of aluminium oxide materials (* - γ-Al_2O_3).

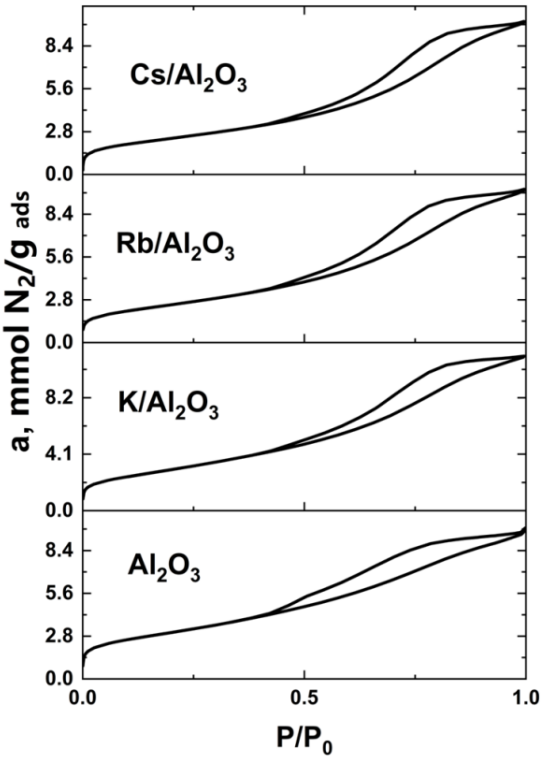

Figure S2 Nitrogen adsorption-desorption isotherms.

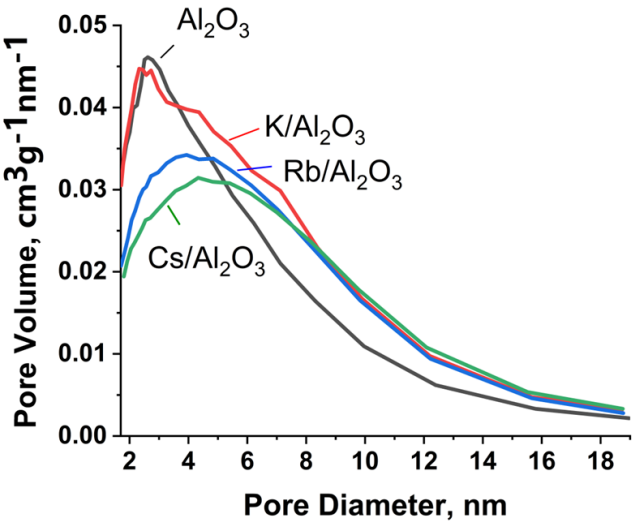

Figure S3 Pore size distribution of the samples.

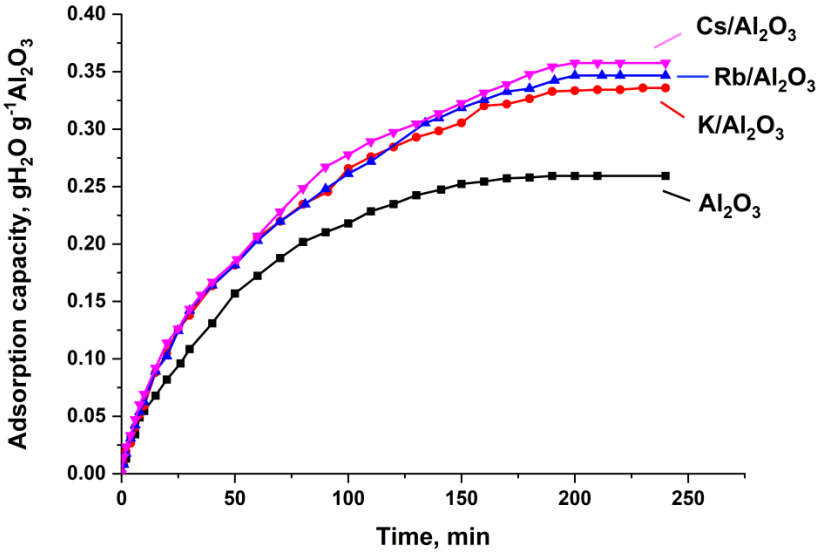

Figure S4 Kinetic curves of water vapour adsorption on the samples for a fraction of 0.5–1.0 mm: Al_2O_3, K/Al_2O_3, Rb/Al_2O_3, Cs/Al_2O_3 (conditions: the carrier gas adsorption rate was 30 L/h).