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Synthesis of LSX zeolites and their N₂ adsorption behavior

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Zeolites belong to a class of inorganic crystalline materials widely used in sorption, catalysis, liquid drying, gas separation and purification. At present, there is a significant demand for high-purity oxygen used in various fields. One of the ways to produce pure oxygen from an air gas mixture is adsorption separation on low-modulus zeolites of the LSX type (low-silica X).

Not only do the separation of air and production of pure O₂ over LSX zeolites occur due to the molecular sieve effect but also due to the ion–quadrupole interaction of extraframework cations (Li⁺, Ca²⁺, Ag⁺) with nitrogen and oxygen molecules of similar size. The LiLSX zeolites were established to possess high nitrogen sorption capacity; they can be used to produce oxygen with a purity of up to 96%. Partial ion exchange of Li⁺ for Ag⁺ can enhance nitrogen adsorption at low pressures (up to 30 kPa) that is important when using zeolites in PSA (pressure swing adsorption).

The aim of this work was to establish the effect of silver loading in AgLiLSX zeolites on room-temperature nitrogen adsorption.

Two series of LSX zeolites with different S_{BET} (434 and 650 m²/g) were prepared. The initial KNaLSX zeolite was synthesized by the sol-gel method followed by crystallization of amorphous alumina-silica gel at 50°C for 72 h. Then, K⁺ and Na⁺ ions were ion exchanged by Li⁺ and then by Ag⁺ ions. Zeolites were studied by XRD, low-temperature N₂ adsorption, UV spectroscopy, atomic emission spectroscopy, and nitrogen adsorption at 25°C.

The formation of zeolites with the LSX structure was confirmed by XRD, all zeolite samples featured a high degree of crystallinity. According to the low-temperature N₂ adsorption-desorption data, it was revealed that all the synthesized samples were microporous. According to the Howarth-Cavazoe method, differential pore size distribution curves indicated that all samples of both series were characterized by micropores of 0.54–0.57 nm in size.

Nitrogen adsorption at room temperature showed that the initial form of the KNaLSX featured a low nitrogen adsorption (3.0–4.5 cm³/g). The LiLSX samples had the highest adsorption capacity to nitrogen, and the maximum adsorption value was determined by the content of Li⁺ (above 15 cm³/g). At low pressures (up to 30 kPa), the AgLiLSX zeolite with the maximum silver content (the maximum degree of exchange of lithium by silver) had the highest N₂ adsorption and characterized by a larger specific surface area than the AgLiLSX of another series.

Thus, the amount of extraframework cations – nitrogen adsorption sites – is determined by S_{BET}; the adsorption properties of the AgLiLSX are affected by the degree of substitution of Li⁺ by Ag⁺. It has been established that the presence of Li⁺ is required for effective nitrogen adsorption on the LSX-type zeolites, and an increase in the silver loading leads to an increase in N₂ adsorption in the low-pressure region.

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