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THERMOGRAVIMETRIC STUDY OF THE SORPTION PROPERTIES OF MODIFIED SILICA GELS

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Abstract

The optimal conditions of the modification process with 2-hydroxyethylcarbamate are defined in the article. Temperature dependence of carbon dioxide sorption on modified silica gels was studied.

Keywords: *silica gel, sorption, carbon dioxide, hydroxyethyl carbamate, thermal stability*

Introduction

The increase in the amount of carbon dioxide in the atmosphere has reached a historical maximum. One of the reasons for this is human activity, as a result of which the amount of gases released into the atmosphere has increased hundreds of times. Deforestation also slows down CO_2 utilization. Scientists have estimated that since the 1850s, half of CO_2 emissions are stored in the atmosphere, but most of it dissolves in ocean waters. Since 2000, the amount of CO_2 in the atmosphere has increased by 12% (Lindsey, 2020; Lüthi D., Le Floch M., Bereiter B., Blunier T., Barnola J. et al., 2008).

Solid sorbents based on amines have attracted the attention of researchers due to their high CO_2 sorption, low energy consumption, high stability, and they do not pollute the environment. Liquid adsorbents have disadvantages, such as high toxicity, foaming, they are not heat-resistant – at high temperatures they undergo destruction and/or evaporation (Geldiev Y. A., Turaev Kh. Kh., Umbarov, 2022; Santana A. M., Freire T. M., Silva L. F., Menezes F. L., Ramos L. L. et al., 2021; Wang F., Yu L. & Huang, 2021).

The main value characterizing the sorption capacity of silica gels that are modified with amino groups is the amount of nitrogen they contain. These sorbents are characterized by maximum sorption kinetics. Since most of the organic amines are on the outer surface, they have been found to fill 70% of the container in 1–10 minutes. It has been shown that the sorption kinetics increases with increasing pore size (Geldiev et al., 2023).

Synhesis method

Synthesis of 2-hydroxyethylcarbamate (HEC). According to the method presented in the literature (Belyaev P. G., Khisamutdinov G. Kh., Sharypova S. G., Konovalova V. P., Kondyukov I. Z., Valeshny S. I., Smirnov S. P., Ilyin, 2008), a mixture of urea and ethanolamine in a 1:1 molar ratio was heated at 110 °C until gas evolution ceased.

10, 20 and 30% solutions of HEC in absolute ethyl alcohol were prepared. Samples of 5.0 g of silica gel were kept in 10.0 g of HEC solution for 2 hours. After this, it was dried at 80 °C to dry mass. The resulting sorbents, depending on the concentration of HEC in the solutions used to modify the silica gel, were designated as S HEC –10, S HEC-20 and S HEC-30.

Thermal analyzes were performed on a Shimadzu TG-600 device at 40–600 °C. To study the kinetics of sorption and desorption, the sorbents were first cleared of absorbed gases and moisture by heating in a nitrogen environment at 110 °C for 30 min, and then kept at 30 °C, 50 °C and 80 °C in a CO_2 environment until constant weight. By repeating the process, it was possible to study the operating cycles of the sorbents.

The degree of modification of the sorbent. The degree of modification of the sorbent in HES solutions was calculated from the increase in its mass. The results of changes in the mass of the samples are given in (table 1).

Table 1. *Results of modification of silica gel with HES*

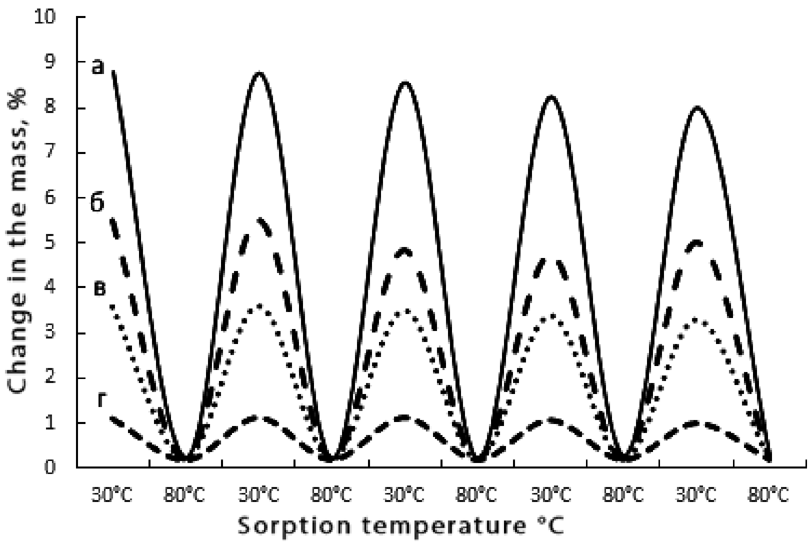
Initial mass of silica gel, g	Composition of solution for HEC/Ethanol modification,%	Received sample	Weight of slick gel after modification	Weight increase,%
5.0	10/90	SHEC –10	6.08	21.6
5.0	20/80	SHEC –20	7.12	42.4
5.0	30/70	SHEC –30	7.76	55.2
5.0	40/60	SHEC –40	7.78	55.6

As can be seen from table. 1, as the concentration of HEC in the solution increased, the degree of modification of the sorbent increased significantly

Results and discussion

Study of the sorption capacity of sorbents. The change in the maximum sorption capacity during sorption/desorption cycles of modified sorbents was studied. Maximum sorption capacities were used at 30 °C for sorption and 80 °C for desorption (Fig. 1). During cyclic operation of modified sorbents in 2 temperature ranges, a decrease of 7–10% was observed after 5 sorption-desorption cycles.

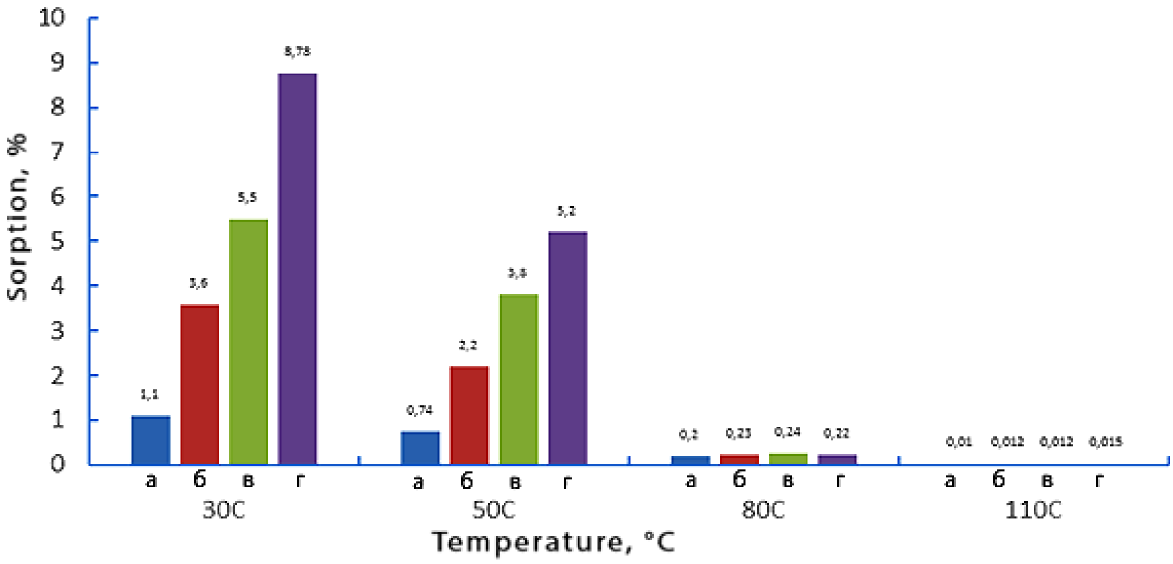
Figure 1. *Cyclic change in the mass of modified silica gels during CO_2 sorption-desorption cycles in comparison with unmodified silica gel. a – SHEK-30; b – SHEC-20; c – SHEC-10; g – SIL*



This decrease can be explained by the partial removal of amino groups at high temperature, since the sorption capacity of the unmodified SIL sample remained virtually unchanged. The optimal temperature for the desorption process was ~80 °C. At a higher temperature, desorption occurred faster and more completely, which led to a significant decrease in the number of effective operating cycles. However, if desorption was carried out at 100 °C, the maximum sorption capacity of the SHEC-30 sample after 30 minutes decreased by more than 30%.

The sorption properties of sorbents significantly depend on the temperature of the gas system. At high temperatures, their CO_2 sorption capacity decreases. CO_2 sorption at 80 °C for modified silica gel is ~0.2%, and at temperatures above 100 °C there is practically no sorption (Fig. 2).

Figure 5. *Temperature dependence of the maximum sorption capacity of sorbents: a – SIL; b – SHEC-10; c – SHEC-20; d – SHEC-30*



The work determined the optimal conditions for modifying silica gel with aqueous solutions of 2-hydroxyethylcarbamate (HEC). Although the surface area of HEC-modified silica gels decreased as the HEC concentration increased, the carbon dioxide sorption capacity increased.

The optimal conditions for the cyclicity of the sorption-desorption process were determined, when sorption occurs at 30 °C and desorption at 80 °C.

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