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LIQUID NITROGEN ADSORPTION ISOTHERM ON AEROSIL

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Abstract

Surface atoms of silicon carrying functional groups may exhibit a tendency to achieve coordination numbers greater than four by forming donor-acceptor bonds with electron-donor molecules; therefore, such atoms are considered active centers. Five-coordinate complexes can be preserved even when the surface is partially filled with isolated hydroxyls. Based on the coordination bond mechanism, adsorbed water molecules are protonated and, as a result, they can also participate as active centers. Their release from the surface occurs in the temperature range of 423–723 K.

Keywords: *Aerosil, liquid nitrogen, adsorption, isotherm*

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Hydrated aerosil is a pure amorphous, nonporous silica consisting of spherical particles of colloidal size and characterized by a large specific surface area (Kiselev, A.V., 1971). During **the SiO_2** formation process, during the drying stage, the particles initially retain their individual shape, and then, under the influence of weak cohesive forces, form primary monodisperse spherical particle aggregates (Zettlemoyer, A. C. Hsing, H.H., Colloid, J., 1977).

Aggregation of particles is facilitated by the presence of water and hydrogen chloride complexes in the surface layer; these substances determine the surface power and hydrogen index (rN) of aerosil hydrogel. During the drying stage, the acid leaves the surface of the particles and the hydrated shell is restored (Snyder, L. Rand, J. W. Ward, J., 1966; Armistead, C.G., and Hockey J. A., 1967; Rehak, V. and Smolkova, E., 1976; Kiselev A. V. and Lygin V. I., 1975). This reduces the force of attraction between particles and prevents their aggregation (Zettlemoyer, A. C. Hsing, H.H., Colloid, J., 1977).

The formation of silanol groups during the formation of aerosil also limits their adhesion to each other during particle growth.

The properties of dispersed silica as an active filler and thickener for dispersion media are determined by the chemical nature, quantity, and geometric arrangement of active centers located in its surface layer. The above-mentioned factors shape the energetic inhomogeneity of the silica surface.

The conducted studies allowed us to determine the chemical nature of the active sites present on the surface of hydrated aerosil. These sites include silicon atoms holding hydroxyl (OH) groups, tight siloxane bridges, silanediol groups, silicon atoms bound to coordinatively unsaturated, isolated hydroxyl groups, and various forms of bound water.

ON groups on the surface of aerosil are of two types: free and hydrogen-bonded. The latter, by its presence, increases the activity of free groups.

Research design and methods

Textural properties such as porosity, specific surface area, and pore size play a key role in determining the effectiveness of aerosils as adsorbents. This section analyzes how different activation methods affect the structure of aerosils and how these changes affect their adsorption properties.

The characteristics of the porous structure (specific surface area –Ssp, total pore volume –VS, pore diameter –dpore) were determined by the low-temperature nitrogen adsorption method at 77 K on a Quantachrome Nova 1000e static-type adsorption device. For this, the studied samples were prepared by vacuum treatment at a temperature of 100 °C for 12 hours. The partial pressure value reached 0.995 P/P0 Nitrogen adsorption/desorption curves were measured in the partial pressure range from 0.005 to 0.995 P/P0. The curves of the dependence of the adsorption amount on the residual pressure were calculated using the BET method.

Research results and discussions

The t-Plot method was used to determine the size of the micropores. The mesopore size was determined using the Barrett-Joyner-Halenda (BJH) method. The average pore diameter was estimated using the BET method using the formula $D_{avg} = 4V/S$. The volume and size distribution of the micropores (average size 1.15–1.17 nm) were estimated using the Horvat-Kawazoe (≈HK) methods.

Nitrogen adsorption/desorption isotherms for carbonates based on licorice root cake are hybrids of type I (in the low relative pressure range; a sharp increase in adsorption at $P/P_0 < 0.05$ is typical of microporous solids) and type IV (in the medium to high pressure range; hysteresis occurs).

The specific surface area reaches a maximum in the temperature range of 400–450 °C, indicating optimal conditions for the development of the porous structure of the cake. At this stage, the maximum specific surface area is approximately 169.8 m²/g.

Temperature above 4 50 °C leads to a decrease in the specific surface area of the mass. This may be due to structural changes such as the coalescence or collapse of pores, which leads to a decrease in the overall porosity.

Then, at a temperature of about 45 0 °C, a secondary increase in specific surface area occurs, which may be the result of further decomposition of the remaining aerosil components and the formation of new pore structures. This secondary increase may also be due to the activation of some types of pores that are inaccessible at lower temperatures due to clogging or blocking by other substances.

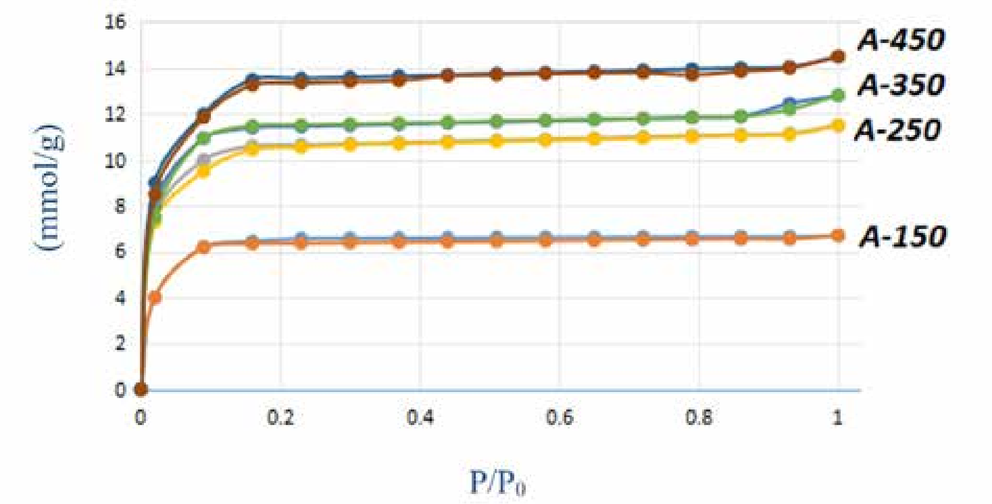
It should be noted that such changes in specific surface area can affect the efficiency of the adsorption properties of aerosil adsorbents. The maximum specific surface area achieved at a given temperature indicates the best conditions for creating an effective adsorbent for a given application.

As can be seen from Figure 1, when the carbonization temperature increases from 150 °C to 450 °C, the micropore volume (V_a) increases, indicating that the development of the microporous structure improves with increasing temperature. The largest micropore volume is observed at 450 °C, which may be the optimal temperature for the formation of a microporous structure in this material.

250 °C, the mesopores occupy the largest volume. This may be due to the initial stage of pore structure formation, when most of the pores have not yet reached microporous sizes. At 450 °C, the mesopore volume decreases, which may indicate the beginning of the pore size reduction and microporous structure formation process. As the temperature increases to 450 °C, the mesopore volume increases again, reaching a maximum in this range, which may indicate the formation of an optimal mesoporous structure.

NaOH solution for activation also significantly increases the porosity of the resulting aerosil. The nature of low-temperature nitrogen adsorption changes, as evidenced by the change in the shape of the isotherm and its values along the y-axis.

Figure 1. *Nitrogen adsorption and desorption isotherms in aerosil: 1) A –150; 2) A –250; 3) A –350; 4) A –450*



Many studies have shown that the activation of aerosil with NaOH leads to an increase in specific surface area, increased porosity, and the development of pore distribution.

All four isotherms resemble IUPAC type IV for mesoporous materials, which are characterized by the presence of a plateau at high relative pressures, indicating the filling of the mesopores.

A –4 5 0 and A –350 show higher adsorption capacity than A –250 and A –150, as the curves for A –450 and A –250 are higher, indicating more adsorbed substance at the same relative pressures. All samples reach saturation (plateau) at relatively low relative pressures, which may indicate high adsorption activity and possibly the presence of a large number of micropores.

The relatively steep initial rise of the curves for all samples indicates the presence of micropores, since the micropores are filled in this pressure range. With increasing relative pressure, the amount of adsorbed substance increases, which is typical of physical adsorption.

Thus, the isotherms show that increasing the amount of activating agent (NaOH) leads to an increase in the adsorption capacity of the activated aerosils. This is consistent with the textural characteristics presented in the table, where the higher ratio of carbonate to NaOH contributed to the higher specific surface area and pore volume.

The textural characteristics of aerosil samples were calculated based on isotherms and are presented in Table 1.

Table 1. *Textural characteristics of aerosil*

Sample	**S_{ud}, m^2/g**	**S_{ud}, m^2/g**	**t – Plot (external surface), m^2/g**	**V_a, cm^3/g**	**V_b, cm^3/g**	**R, Å**	**Average pore width, Å**	**Average hydraulic radius of pores, Å**
A –150	360.17	578.13	78.87	0.2147	0.2478	21.971	127.11	6.895
A –250	517.67	879.17	166.89	0.3784	0.3787	22,243	90.78	6,778
A –350	778.57	1278.47	185.26	0.4128	0.4841	22.347	65.74	6.658
A –450	897.17	1347.21	203.36	0.4974	0.5871	22.585	45.47	6.624

The specific surface area increases from A- 350 to A –450, which may be due to the increase in the amount of activating agent (NaOH). This increase in surface area contributes to the increase in adsorption capacity, as evidenced by the adsorption isotherms. However, A –350 has a slightly lower specific surface area than A –450, which may indicate a certain threshold, after which further increase in NaOH does not lead to a further increase in specific surface area due to excessive erosion or pore clogging. The volume of micro (V_a) and mesopores (V_b) also increases with increasing flow rate of NaOH solution.

The conclusion of this section shows the importance of temperature and carbonization time in the formation of the porous structure of licorice root cake-based aerosils. Studies have shown that with increasing carbonization temperature, the specific surface area of the material changes and reaches a maximum in the temperature range of 400–450 °C, which indicates optimal conditions for the development of a porous structure. However, a further increase in temperature above 450 °C leads to a decrease in the specific surface area due to structural changes such as pore coalescence or collapse.

Aerosils treated at different temperatures, showing higher porosity and adsorption activity in samples treated at the optimal temperature range. These data are important for understanding the mechanisms of pore formation and the subsequent influence of these pores on the adsorption properties of aerosils.

Thus, the results indicate that controlling the temperature conditions for aerosil regeneration above 400^0 Cis crucial for optimizing the textural properties of aerosils and can significantly improve their adsorption properties. These findings will help in the development of aerosil adsorbents with predictable performance and high efficiency, which is the key to their successful industrial application.

The formation of porous structures and the change in the chemical bonding of aerosil emphasize the transformation of the material into a more active form, which increases the adsorption properties.

Analysis of nitrogen adsorption and desorption isotherms confirmed a significant improvement in the textural properties of the aerosilnong. The increase in specific surface area and pore volume was attributed to the removal of unwanted organic components and the formation of new micro- and mesopores. The optimal activation temperature and duration were the key factors for achieving maximum porosity and adsorption capacity.

Conclusion

The observed changes in the surface chemistry and adsorption properties of silanized samples compared to the same dehydrated samples indicate that the silanization process is accompanied by the development of surface porosity. As the activity of silanols on the surface increases, the surface porosity increases significantly. It is shown that the properties of aerosil can be significantly improved and make them suitable for use in various industrial applications requiring high-performance adsorbents. These findings will help to develop new approaches to create aerosil adsorbents with predictable and improved performance.

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