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SORPTION PROPERTIES OF SYNTHETIC OH- AND Cl-FORMS OF SODALITE

Abstract. This paper presents the studies of argon, methanol and water vapour sorption on Cl- and OH-forms of sodalite. The volume of respective adsorbates required to coat the surface of sodalite with a monomolecular layer was determined, and the specific surface area was calculated for the two forms of sodalite. It was found that the sorption properties of sodalite depend on the initial material used for synthesis. The samples were subjected to thermal activation, and the resulting changes in sorption properties were discussed. The binding mechanism of molecules of different adsorbates to the surface of sodalites was studied.

INTRODUCTION

As a continuation of the earlier studies of crystallochemical properties of synthetic OH- and Cl-sodalite (Tokarz 1978; Fijał, Tokarz 1981), the sorption properties of this mineral were investigated. There are very few papers dealing with this subject because such investigations have hardly ever been carried out on sodalites. The reason for this is presumably the fact that the "windows" in the structure of sodalite have a small diameter (about 1.5 and 2.3 Å), which limits severely the possibility of incorporation of large-size adsorbate molecules into this structure. It seems, however, that sodalite could be used as a selective molecular sieve to separate small molecules.

EXPERIMENTAL

The investigations were carried out on some of the samples studied and described earlier by Fijał and Tokarz (1981). These were OH- and Cl-sodalites obtained by synthesis under hydrothermal conditions from pure chemical components, and through transformation of kaolinite. In the latter case, kaolinite was derived from the Jęglowa and Kalno deposits. The list of samples and the general conditions of synthesis are given in Table 1. Additional information on the synthesis conditions can be found in the papers of Tokarz (1978) and Fijał, Tokarz (1981).

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Table 1

Sodalite samples investigated for sorption properties

Sample symbol	Kind of nonstoichiometric ions		Starting materials
<i>S-16</i>	Na ⁺	Cl ⁻	Silica sol + sodium aluminate + NaOH + NaCl + H ₂ O
<i>S-17</i>	Na ⁺	OH ⁻	Silica sol + sodium aluminate + NaOH + H ₂ O
<i>S-41</i>	Na ⁺	OH ⁻	Kaolinite (Jęglowa) + NaOH + H ₂ O
<i>K-3</i>	Na ⁺	OH ⁻	Kaolinite (Kalno) + NaOH + H ₂ O
<i>K-14</i>	Na ⁺	Cl ⁻	Kaolinite (Kalno) + NaOH + NaCl + H ₂ O

Sorption isotherms were determined using volumetric equipment. Sorption manostats (Ciembroniewicz, Lasoń 1972) were used to determine argon sorption and desorption isotherms at the liquid nitrogen temperature, i.e. at 77.5 K. At this temperature the pressure of saturated argon vapour is 2.66 Pa. The isotherms for water and methanol sorption were obtained at 298 K using microburettes for liquids (Lasoń, Żyła 1963). At this temperature the pressure of saturated water vapour and methanol is 0.316 Pa and 1.64 Pa, respectively.

The choice of adsorbates was determined both by the kinetic diameter of their molecules and the degree of their polarity. Due to this, it was possible to estimate the part of physical adsorption and chemisorption in the total amount of sorption. Physical adsorption takes place as a result of non-specific interaction between the adsorbent surface and non-polar argon molecules, whereas chemisorption is caused by the interaction between the active acid-basic surface centres and polar adsorbate molecules.

From the isotherms obtained for argon, methanol and water vapour adsorption the number of adsorbate molecules necessary to coat the adsorbent surface with a monomolecular layer (V_m) was calculated using the BET equation (Brunauer *et al.* 1938). Then the specific surface area was determined for the samples studied, using the formula:

$$S = V_m \cdot s$$

where: S — specific surface area of adsorbent, m²/g,

V_m — number of adsorbate molecules in monolayer,

s — surface area occupied by one adsorbate molecule, Å².

The value of specific surface area obtained by means of a polar adsorbate for adsorbents that have active centres on their surface, is questionable. In such a case, the amount of sorbed molecules corresponds to the number of active centres rather than being a direct measure of specific surface area. It is to be assumed that the V_m value in the BET equation gives then information on the degree of saturation of the polar centres on the adsorbent surface, but does not say whether a monomolecular adsorbate layer has formed on this surface. When the number of polar centres on the adsorbent surface increases markedly, the specific surface area calculated from the sorption of polar substances is close to the actual value.

Table 2

Sorption investigations for OH⁻ and Cl⁻-sodalite

Sample symbol	Argon				Methanol				Water			
	V_m , cm ³ /g		S_{BET} , m ² /g		V_m , m ³ /g		S_{BET} , m ² /g		V_m , cm ³ /g		S_{BET} , m ² /g	
	393 K	593 K	393 K	593 K	393 K	593 K	383 K	593 K	393 K	593 K	393 K	593 K
<i>K-3</i>	$0.6 \cdot 10^{-2}$	$0.6 \cdot 10^{-2}$	20.7	20.7	$3.06 \cdot 10^{-2}$	$1.61 \cdot 10^{-2}$	82.0	43.2	$2.26 \cdot 10^{-2}$	$7.38 \cdot 10^{-2}$	79.4	259.3
<i>S-41</i>	$0.3 \cdot 10^{-2}$	$0.3 \cdot 10^{-2}$	10.1	10.1	$2.12 \cdot 10^{-2}$	$0.6 \cdot 10^{-2}$	56.8	15.8	$1.84 \cdot 10^{-2}$	$5.29 \cdot 10^{-2}$	64.7	187.5
<i>K-14</i>	$0.23 \cdot 10^{-2}$	—	7.9	—	—	$0.34 \cdot 10^{-2}$	—	9.1	$0.47 \cdot 10^{-2}$	$0.66 \cdot 10^{-2}$	16.6	23.1
<i>S-16</i>	—	$0.08 \cdot 10^{-2}$	—	2.8	—	$2.32 \cdot 10^{-2}$	—	62.2	—	$0.62 \cdot 10^{-2}$	—	21.9

Sorption measurements also help to determine the structure of pores. This structure can be described by determining the pore volume and the corresponding effective radius. Recent studies have confirmed that such calculations are more accurate if the 2nd variant of Dubinin-Raduszkiewicz method is adopted (Ościak 1978).

RESULTS

Measurements of argon, methanol and water vapour sorption were carried out on synthetic OH- and Cl-sodalites. The samples were outgassed for 12 hours at 393 and 593 K at a vacuum of 10^{-5} mm Hg. The results are presented in the form of isotherm in Figures 1—6 and in Table 2.

The K-3 sample, representing the OH-form of sodalite obtained by way of hydrothermal transformation of kaolinite from the Kalno deposit, shows the best sorption

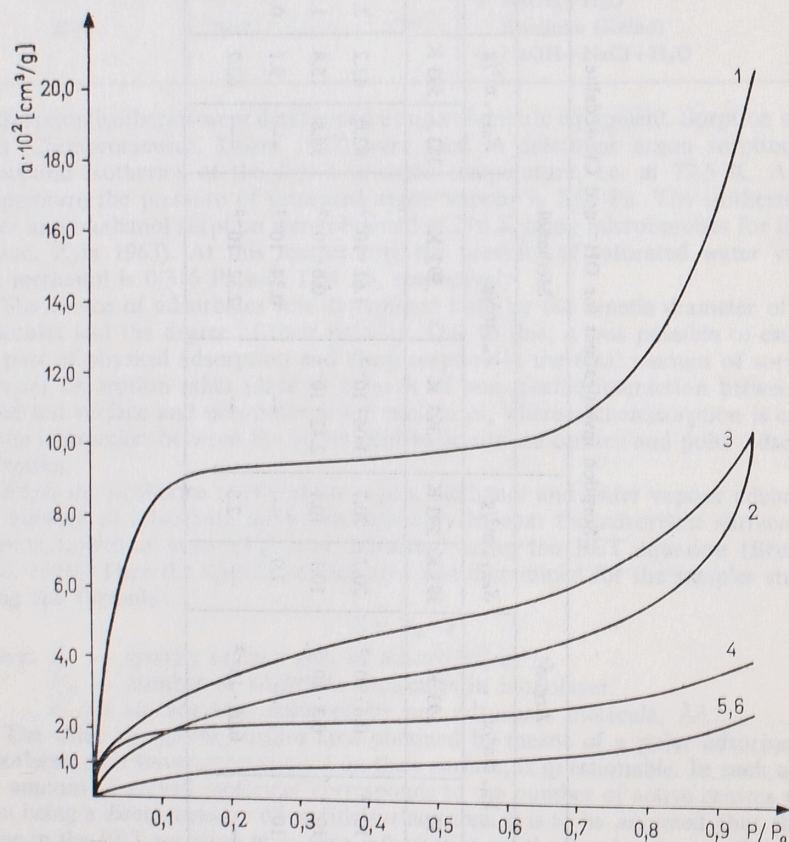


Fig. 1. Sorption isotherms of argon, water vapour and methanol by OH-form of sodalite — sample K-3 (in parentheses the outgassing temperature of the sample is given)

1 — H₂O (593 K), 2 — H₂O (393 K), 3 — CH₃OH (393 K), 4 — CH₃OH (593 K), 5 — argon (593 K), 6 — argon (393 K)

properties of all the samples studied. As appears from the argon, methanol and water vapour sorption isotherms (Fig. 1), water vapour sorption in the initial range of relative pressures is about 4.5 times higher than methanol sorption. This difference increases to about 5 times for relative pressures close to the pressure of saturated vapours. The adsorbate volume V_m in the monolayer is 7.38×10^{-2} cm³/g for water vapour and 1.61×10^{-2} cm³/g for methanol.

When the sample was outgassed at 393 K, the shape of adsorption curves is similar to that of type II isotherms according to the BET classification (Ościak 1979). At relative pressures $p/p_0 = 0.0-0.9$ such samples show methanol sorption higher by $1.0-1.5 \times 10^{-2}$ cm³/g than water vapour sorption. However, under the conditions of maximum saturation with adsorbate vapours ($p/p_0 = 0.95$), the amounts of adsorbed water vapour and methanol are nearly the same, being close to 0.1 cm³/g.

Argon sorption of the K-3 sample is the lowest. The adsorbate volume in the monolayer is 0.6×10^{-2} cm³/g. From the shape of the argon sorption isotherm it appears that there is no specific interaction between the non-polar adsorbate and the adsorbent surface.

The S-41 sample represents the same OH-form of sodalite but the initial kaolinite used for synthesis was derived from the Jegłowa deposit. The general shape of sorption isotherms (Fig. 2) is similar to that of the isotherms obtained for the K-3 sample. The only differences have been noted in the initial range of relative pressures. The slopes of isotherms are less steep, indicating that the number of adsorbent-adsorbate ionic bonds has decreased. The amount of water vapour adsorbed by the sample outgassed at 593 K is 6—7 times greater than the amount of adsorbed

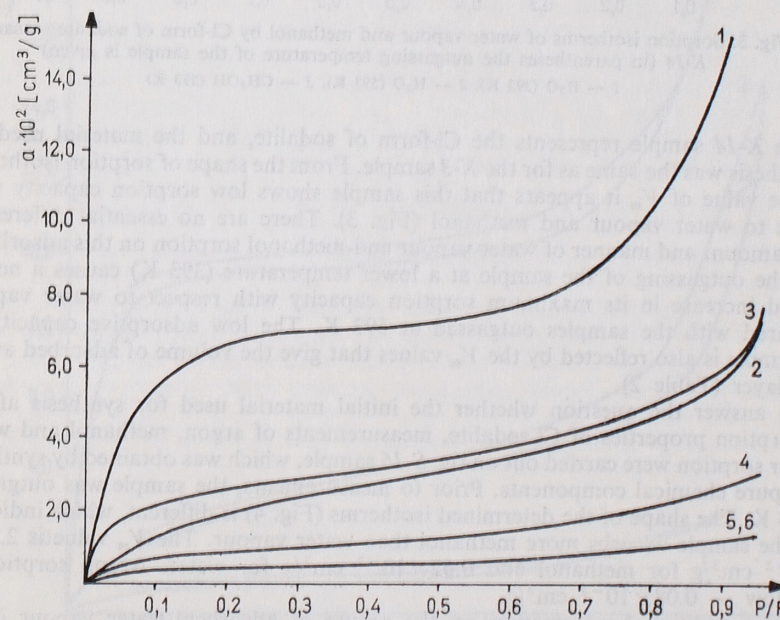


Fig. 2. Sorption isotherm of argon, water vapour and methanol by OH-form of sodalite — sample S-41 (in parentheses the outgassing temperature of the sample is given)

1 — H₂O (593 K), 2 — H₂O (393 K), 3 — CH₃OH (393 K), 4 — CH₃OH (593 K), 5 — argon (593 K), 6 — argon (393 K)

methanol over the whole range of relative pressures. The values of V_m are 5.29×10^{-2} and 0.6×10^{-2} cm³/g, respectively. As was the case in the *K-3* sample, the low-temperature argon sorption is insignificant (Fig. 2), and the value of V_m is 0.3×10^{-2} cm³/g. This value does not depend on the outgassing temperature, being the same for temperatures of 393 and 593 K.

The *S-17* sample was obtained by synthesis from pure chemical components. From the shape of sorption isotherms determined for this sample it is impossible to establish with confidence the equilibrium relative pressure at which the adsorbent surface becomes completely saturated by the monomolecular layer of adsorbed water vapour. The amounts of water adsorbed at a given relative pressure p/p_0 lie between the respective values obtained for the samples *K-3* and *S-41*. The value of V_m for the *S-17* sample is 5.53×10^{-2} cm³/g.

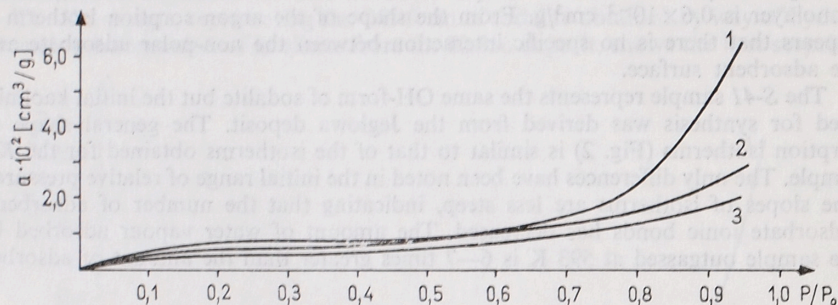


Fig. 3. Sorption isotherms of water vapour and methanol by Cl-form of sodalite — sample *K-14* (in parentheses the outgassing temperature of the sample is given)
1 — H₂O (393 K), 2 — H₂O (593 K), 3 — CH₃OH (593 K)

The *K-14* sample represents the Cl-form of sodalite, and the material used for its synthesis was the same as for the *K-3* sample. From the shape of sorption isotherms and the value of V_m it appears that this sample shows low sorption capacity with respect to water vapour and methanol (Fig. 3). There are no essential differences in the amount and manner of water vapour and methanol sorption on this adsorbent. Only the outgassing of the sample at a lower temperature (393 K) causes a nearly twofold increase in its maximum sorption capacity with respect to water vapour compared with the samples outgassed at 593 K. The low adsorptive capacity of this sample is also reflected by the V_m values that give the volume of adsorbed argon monolayer (Table 2).

To answer the question whether the initial material used for synthesis affects the sorption properties of Cl-sodalite, measurements of argon, methanol and water vapour sorption were carried out on the *S-16* sample, which was obtained by synthesis from pure chemical components. Prior to measurements, the sample was outgassed at 593 K. The shape of the determined isotherms (Fig. 4) is different, which indicates that the sample adsorbs more methanol than water vapour. The V_m value is 2.32×10^{-2} cm³/g for methanol and 0.62×10^{-2} cm³/g for water. Argon sorption is very low — 0.08×10^{-2} cm³/g.

Some additional information on the nature of adsorbent-water vapour interaction is provided by the sorption-desorption curves (Fig. 5). The hysteresis loops are open, indicating that a certain amount of water is strongly bound to the adsorbent surface and fails to be removed from the surface of sodalite under the desorption conditions used.

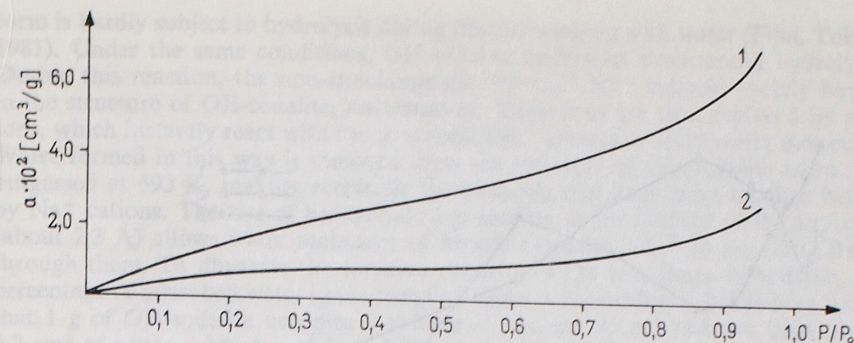


Fig. 4. Sorption isotherms of water vapour and methanol by Cl-form of sodalite — sample *S-16* outgassed at 593 K
1 — CH₃OH, 2 — H₂O

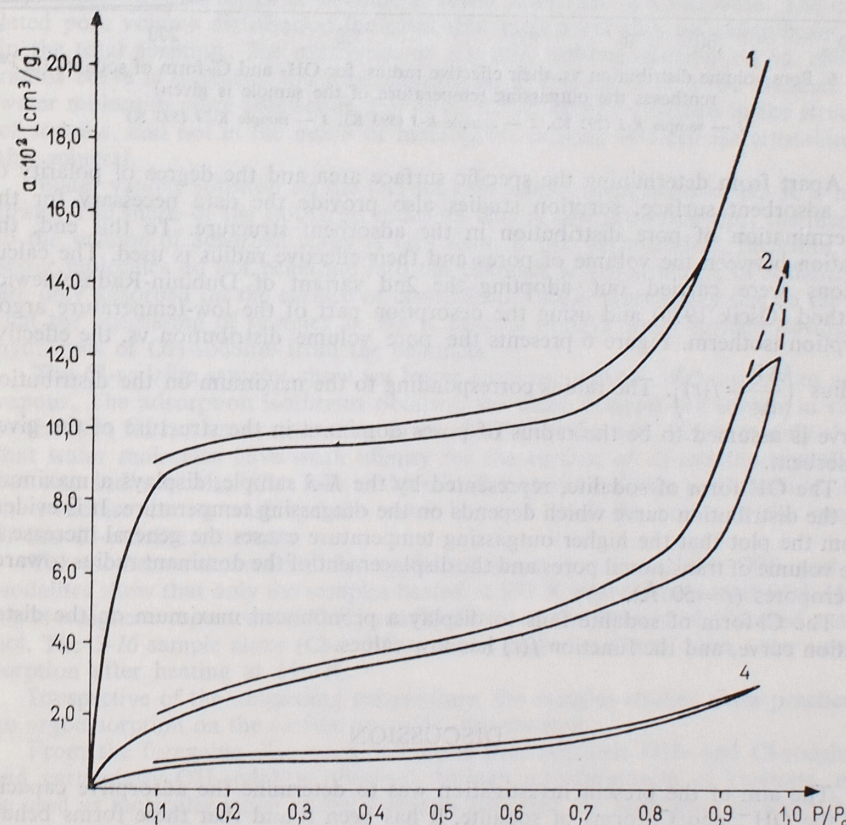


Fig. 5. Sorption and desorption isotherms of water vapour — samples *K-3* and *K-14* (in parentheses the outgassing temperature of the sample is given)
1 — sample *K-3* (593 K), 2 — sample *K-3* (393 K), 3 — sample *K-14* (593 K)

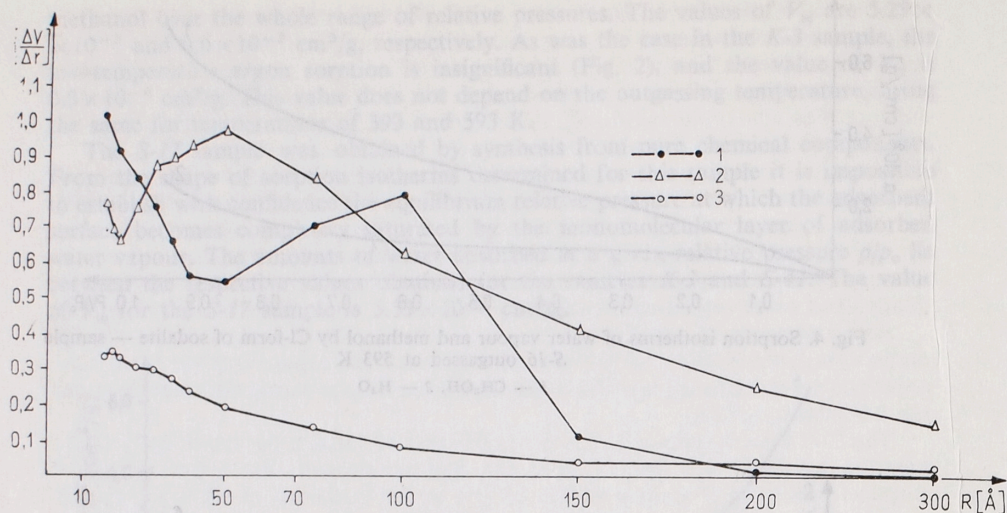


Fig. 6. Pore volume distribution vs. their effective radius, for OH- and Cl-form of sodalite (in parentheses the outgassing temperature of the sample is given)

1 — sample K-3 (393 K), 2 — sample K-3 (593 K), 3 — sample K-14 (593 K)

Apart from determining the specific surface area and the degree of polarity of the adsorbent surface, sorption studies also provide the data necessary for the determination of pore distribution in the adsorbent structure. To this end, the relation between the volume of pores and their effective radius is used. The calculations were carried out adopting the 2nd variant of Dubinin-Raduszkiewicz method (Ościak 1978) and using the desorption part of the low-temperature argon sorption isotherm. Figure 6 presents the pore volume distribution vs. the effective radius $\left(\frac{\Delta V}{\Delta r} = f(r)\right)$. The radius corresponding to the maximum on the distribution curve is assumed to be the radius of pores dominant in the structure of the given adsorbent.

The OH-form of sodalite, represented by the K-3 sample, displays a maximum on the distribution curve which depends on the outgassing temperature. It is evident from the plot that the higher outgassing temperature causes the general increase in the volume of transitional pores and the displacement of the dominant radius towards micropores ($r = 50 \text{ Å}$).

The Cl-form of sodalite fails to display a pronounced maximum on the distribution curve, and the function $f(r)$ has low values.

DISCUSSION

The aim of the present investigation was to determine the adsorptive capacity of the OH- and Cl-forms of sodalite. It has been found that these forms behave differently with respect to the adsorbates used. The majority of OH-sodalite samples show far better sorption properties with respect to water vapour than Cl-sodalites. It seems that the low sorption capacity of Cl-sodalite is due to the fact that this

form is hardly subject to hydrolysis during intense washing with water (Fijał, Tokarz 1981). Under the same conditions, OH-sodalite undergoes pronounced hydrolysis. During this reaction, the non-stoichiometric "excess" Na^+ cations, loosely bound in the structure of OH-sodalite, are removed. These ions are then replaced by protons, which instantly react with the structural OH^- groups to form water molecules. Water formed in this way is removed from the structure of OH-sodalite when it is outgassed at 593 K, making accessible the channels that have been blocked before by Na^+ cations. The size of hexagonal rings leading to the interior of the structure (about 2.3 Å) allows water molecules of kinetic diameter 2.2 Å to penetrate freely through them. To illustrate the sorption capacity of the two forms of sodalite, the percentage of adsorbed water in the sample volume was calculated. It has been found that 1 g of OH-sodalite occupies a volume of about 0.45 m^3 and can bind about 0.2 cm^3 of water, while 1 g of Cl-sodalite binds only 0.028 cm^3 of water, occupying the same volume. In the initial range of relative pressures $p/p_0 = 0.0-0.1$, the rapid increase in the amount of adsorbed water has been noted for all the OH-sodalite samples. Such a shape of isotherms is typical of the sorption in micropores. From X-ray structural studies (Fijał, Tokarz 1981) it follows that the degree of order and crystallinity of OH-forms of sodalite is lower than that of Cl-sodalite. The calculated pore volume distribution indicates that mesopores play an insignificant part in the total sorption. The maximum on the pore volume distribution vs. effective radius curve is displaced towards micropores. Therefore, it is to be assumed that water molecules place themselves first of all in the narrow channels in the structure of sodalite, and not in the meso- or macropores existing between the crystallites of this mineral.

Water vapour sorption on the OH-sodalite samples outgassed at 393 K is much lower. The shape of the isotherms indicates that at low pressures p/p_0 , the increase in the volume of adsorbed water is very slow. It can be presumed, therefore, that water molecules do not penetrate into the channels in the structure of sodalite but are only adsorbed on the surface of crystallites. This is presumably due to the fact that the outgassing of samples at 393 K fails to remove water formed during the hydrolysis of OH-sodalite from the channels.

The Cl-sodalite samples show yet lower sorption capacity with respect to water vapour. The adsorption isotherms obtained for these samples are similar in shape to the type III isotherms according to the BET classification. This not only shows that water molecules have weak affinity for the surface of Cl-sodalite crystallites, but also indicates that Cl^- ions of kinetic diameter 1.8 Å are not removed from the structure during washing with water but still block the access to the interior of the Cl-sodalite structure.

The isotherms determined for methanol sorption on synthetic OH- and Cl-sodalites show that only the samples heated at 393 K adsorb some methanol. Heating at a higher temperature (593 K) usually decreases the amount of adsorbed methanol. The S-16 sample alone (Cl-sodalite) shows higher methanol than water vapour sorption after heating at 593 K.

Irrespective of the outgassing temperature, the samples studied show practically no argon sorption on the surface or inside the structure.

From the foregoing discussion it follows that synthetic OH- and Cl-sodalites, and particularly OH-sodalites obtained through transformation of kaolinite, may be used as molecular sieves in the processes requiring the separation of water from organic compounds.

CONCLUSIONS

1. The OH-form of sodalite has definitely better sorption properties than the Cl-form. This is due to the hydrolysis of OH-sodalite, which renders additional channels in the structure of this mineral accessible to sorption.

2. There is a marked correlation between the outgassing temperature of OH-sodalite and its adsorptive capacity with respect to water vapour. Water adsorbed by OH-sodalite may occupy up to 45% of its initial volume.

3. The sorption properties of OH-sodalite depend on the provenance of kaolinite used for its synthesis.

4. The Cl-sodalite sample obtained by synthesis from pure chemical components shows higher sorption capacity with respect to methanol than the sample obtained from natural kaolinite. Both samples have similar water sorption capacity.

5. The pore volume distribution vs. effective pore radius curve plotted for OH-sodalite displays a maximum corresponding to the dominant radius $r=100$ Å. The outgassing of the sample at 593 K results in the general increase in the volume of transitional pores and causes the dominant radius to shift towards micropores ($r=50$ Å).

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WŁASNOŚCI SORPCYJNE SYNTETYCZNEGO OH- I Cl-SODALITU

Streszczenie

Wykonano badania chłonności sorpcyjnych formy Cl- i OH-sodalitów względem argonu oraz par metanolu i wody. Wyznaczono objętości poszczególnych adsorbatów potrzebne do pokrycia powierzchni sodalitu warstwą monomolekularną oraz obliczono powierzchnię właściwą poszczególnych jego form. Stwierdzono zależność własności sorpcyjnych sodalitu od materiału wyjściowego który został użyty do syntezy. Przeprowadzono termiczną aktywację próbek i wykazano zmiany ich własności sorpcyjnych. Przeanalizowano mechanizm połączeń cząsteczek stosowanych adsorbatów z powierzchnią różnych form sodalitu.

OBJAŚNIENIA FIGUR

- Fig. 1. Izotermy adsorpcji argonu, par wody i metanolu przez OH-formę sodalitu — próbka K-3 (w nawiasach podano temperaturę odgazowania próbki)
 1 — H₂O (593 K), 2 — H₂O (393 K), 3 — CH₃OH (393 K), 4 — CH₃OH (593 K), 5 — argon (593 K), 6 — argon (393 K)
 Fig. 2. Izotermy adsorpcji argonu, par wody i metanolu przez OH-formę sodalitu — próbka S-41 (w nawiasach podano temperaturę odgazowania próbki)
 1 — H₂O (593 K), 2 — H₂O (393 K), 3 — CH₃OH (393 K), 4 — CH₃OH (593 K), 5 — argon (593 K), 6 — argon (393 K)
 Fig. 3. Izotermy adsorpcji par wody i metanolu przez Cl-formę sodalitu — próbka K-14 (w nawiasach podano temperaturę odgazowania próbki)
 1 — H₂O (393 K), 2 — H₂O (593 K), 3 — CH₃OH (593 K)
 Fig. 4. Izotermy adsorpcji par wody i metanolu przez Cl-formę sodalitu — próbka S-16 odgazowywana w temperaturze 593 K
 1 — CH₃OH, 2 — H₂O
 Fig. 5. Izotermy adsorpcji i desorpcji par wody przez próbki K-3 i K-14 (w nawiasach podano temperaturę odgazowania próbki)
 1 — próbka K-3 (593 K), 2 — próbka K-3 (393 K), 3 — próbka K-14 (593 K)
 Fig. 6. Funkcja rozkładu objętości porów względem ich efektywnych promieni dla OH- i Cl-form sodalitu (w nawiasach podano temperaturę odgazowania próbki)
 1 — próbka K-3 (393 K), 2 — próbka K-3 (593 K), 3 — próbka K-14 (593 K)

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ПОГЛОЩАЮЩИЕ СВОЙСТВА ИСКУССТВЕННОГО OH- И Cl-SОДАЛИТА

Резюме

Проведено исследования поглощающих свойств модификаций Cl- и OH-содалитов в отношении аргона, а также паров метанола и воды. Определено объемы отдельных абсорбатов, необходимые для покрытия поверхности содалита молекулярным слоем, а также рассчитано удельную поверхность отдельных его форм. Обнаружено зависимость поглощающих свойств содалита от исходного материала, который применялся в синтезе. Проведено термическую активацию образцов и доказано изменение их поглощающих свойств. Проанализирован механизм соединений молекул употребляемых адсорбатов с поверхностью различных форм содалита.

ОБЪЯСНЕНИЯ К ФИГУРАМ

- Фиг. 1. Изотермы адсорбции аргона, паров воды и метанола на OH-модификации содалита, образец K-3 (в скобках указана температура дегазации образца)
 1 — H₂O (593 K), 2 — H₂O (393 K), 3 — CH₃OH (393 K), 4 — CH₃OH (593 K), 5 — аргон (593 K), 6 — аргон (393 K)
 Фиг. 2. Изотермы адсорбции аргона, паров воды и метанола на OH-модификации содалита, образец S-41 (в скобках указана температура дегазации образца)
 1 — H₂O (593 K), 2 — H₂O (393 K), 3 — CH₃OH (393 K), 4 — CH₃OH (593 K), 5 — аргон (593 K), 6 — аргон (393 K)

Фиг. 3. Изотермы адсорбции паров воды и метанола на СI-модификации содалита, образец *K-14* (в скобках указана температура дегазации образца)

1 — H_2O (393 К), 2 — H_2O (593 К), 3 — CH_3OH (593 К)

Фиг. 4. Изотермы адсорбции паров воды и метанола на СI-модификации содалита, образец *S-16*, дегазированный в температуре 593 К

1 — CH_3OH , 2 — H_2O

Фиг. 5. Изотермы адсорбции и десорбции паров воды на образцах *K-3* и *K-14* (в скобках указана температура дегазации образца)

1 — образец *K-3* (593 К), 2 — образец *K-3* (593 К), 3 — образец *K-14* (593 К)

Фиг. 6. Функция распределения объема паров по отношению к их эффективным радиусам для ОН- и СI-модификаций содалита (в скобках указана температура дегазации образца)

1 — образец *K-3* (393 К), 2 — образец *K-3* (593 К), 3 — образец *K-14* (593 К)

Проведено исследование пористых материалов, модифицированных СI- и ОН- группами в отношении их способности адсорбировать пары воды и метанола. В работе описаны методы исследования, результаты которых показывают, что модифицированные материалы обладают высокой способностью к адсорбции паров воды и метанола. В частности, для образцов *K-3* и *K-14* (СI-модификация) и *S-16* (ОН-модификация) были получены изотермы адсорбции и десорбции паров воды. Результаты показывают, что модифицированные материалы обладают высокой способностью к адсорбции паров воды и метанола. В частности, для образцов *K-3* и *K-14* (СI-модификация) и *S-16* (ОН-модификация) были получены изотермы адсорбции и десорбции паров воды. Результаты показывают, что модифицированные материалы обладают высокой способностью к адсорбции паров воды и метанола.