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## ISOTHERM OF AMMONIA ADSORPTION IN ZEOLITE CaA (M-22)

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#### Abstract:

**Objective.** The article presents experimentally obtained values of the adsorption isotherm of ammonia molecules in synthetic zeolite CaA (M-22) at a temperature of 303 K. Isotherm values were measured using an improved microcalorimeter connected to a universal high vacuum apparatus. The differential values of the free energy were calculated from the equilibrium values of the pressure. In CaA (M-22) zeolite, a regular relationship between the amount of adsorption and energy properties of ammonia molecules, as well as the sorption mechanism from the initial area of adsorption to the area of condensation heat of ammonia and the regularity of ammonia molecules filling the volume of zeolite were determined. Under experimental conditions, the adsorption capacity of this zeolite for ammonia was found to be 10.5 mmol/g in 1 g of zeolite. It was determined that 40% of the total adsorption is sorbed up to the equilibrium pressure of 1 torr, 50% up to 6 torr pressure, 60% up to 24 torr pressure, 65% up to 50 torr pressure and 100% at 467 torr pressure. The adsorption isotherm was recharacterized by the three-state equation of micropore volumetric saturation theory (VMOT) and it was shown that the theoretically calculated values were in perfect agreement with the experimentally obtained values.

**Methods.** The adsorption isotherm was measured with high accuracy (enthalpy 0.2  $\mu$ J, small values of pressure with an accuracy of  $10^{-5}$  torr) and stability using a system consisting of a Tian-Calve type DAK-1-1A differential automated microcalorimeter connected to a universal high-vacuum device. The adsorption-calorimetric method used in the research allows obtaining molar thermodynamic characteristics, as well as revealing the detailed mechanisms of adsorbent-adsorbate and adsorbate-adsorbate sorption processes. Adsorption measurements and dosage of adsorbate were performed using a high-vacuum adsorption device. The device allows dosing of adsorbate by gas-volume and volume-liquid methods. B627 diaphragm baratron was used to measure the equilibrium pressure up to  $10^{-5} \pm 0,8$  torr, U manometer was used to measure the pressures at  $R > 0,8$  torr. The adsorption-calorimetric method allows to study nano-, micro-, mesostructured adsorbents and their surface-active surfaces, to reveal in detail the main thermodynamic properties and mechanisms of adsorption processes in which adsorbents occur.

**Results.** Adsorption of ammonia molecules on CaA (M-22) zeolite at a temperature of 303 K from the area of small saturations to the heat of condensation of ammonia from the initial area to the saturation pressure was found that the enthalpy values of ammonia adsorption in this zeolite are almost 40-50 kJ/mol higher than the adsorption enthalpy of polar and non-polar molecules of different nature. During the adsorption process, it was found that ammonia molecules interact with  $\text{Na}^+$  and  $\text{Ca}^{2+}$  cations in  $\text{S}_I$  and  $\text{S}_{II}$  positions of zeolite.

**Conclusion.** The results of adsorption-calorimetric research obtained on the basis of experience allow to obtain the main thermodynamic functions of the studied systems, which are necessary for the

development of theoretical concepts of chemical and physical adsorption in synthetic zeolites of the LTA type, including CaA, as well as in the calculation of sorption technology processes and devices in practice.

**Keywords:** adsorption, enthalpy, free energy, isotherm, pressure, relative pressure, microcalorimeter, ammonia.

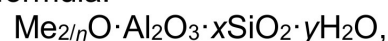
**Introduction.** In order to obtain environmentally friendly gas from natural gases, the demand for drying them from water vapor, cleaning them from gases such as hydrogen sulfide, and preventing environmental problems caused by the release of greenhouse gases, including carbon (IV) oxide into the atmosphere, is increasing year by year. For this purpose, synthetically produced zeolites are widely used to avoid the above-mentioned problems. It is important to achieve scientific and practical innovations based on the results of the research conducted on the synthesis of zeolites with high adsorption and catalytic properties, increasing the level of their selectivity.

Today, in the world, the following scientific solutions for the synthesis of nanoporous molecular bubble zeolites for adsorption processes are justified, including: the selection of raw materials with suitable adsorption properties to obtain zeolites with selective absorption properties; determination of complete thermodynamic properties of synthesized zeolites; formation of ion-molecular complexes in the zeolite matrix, determining the state of localization; studying thermokinetics of adsorption and cation exchange in the zeolite structure, migration of cations; the complete molecular mechanism of zeolite adsorption should be determined.

Aluminum and silicon atoms in aluminosilicate zeolites can be replaced by 3-5 valence elements such as gallium, germanium, phosphorus, which are close to them in nature, and their sorption and catalytic properties can be changed [1]. Another characteristic of these zeolites is the presence of water molecules in the internal structure of the crystal, when heated to a temperature of 450°C, water molecules evaporate without breaking the

structure of the crystal lattice, and the possibility of ion exchange due to the mobility of alkaline, alkaline earth cations and water molecules in the zeolite [2-4].

Aluminosilicate zeolites with a porous crystal framework structure. The frameworks of zeolites are composed of interconnected tetrahedral [SiO<sub>4</sub>] and [AlO<sub>4</sub>], the ends of which are connected by oxygen atoms. The unique arrangement of aluminum atoms in the structure is one of the properties of aluminosilicates. Al and Si atoms are arranged in the tetrahedral coordination position in all aluminosilicates with respect to oxygen and are isomorphically substituted for silicon in the general silicon-oxygen framework. The negative charge of the tetrahedron is neutralized by various alkaline or alkaline earth positive cations located in the zeolite voids. The composition of all synthetic zeolites can be described by the following chemical formula:



where n-metal cation valency, x-SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> molar ratio, y-number of moles of water.

Based on the structure and composition of the crystal lattice of zeolites, zeolites are characterized by the names of MFI, MOR, FAU, LTA, regardless of their highest symmetry, which can choose sorption properties and catalysis characteristics [5] and their main thermodynamic properties of catalysis and adsorption of polar, non-polar and quadrupole molecules of different nature are fundamentally different from each other [6-11].

MFI, MOR, FAU, LTA and other types of zeolites form adsorbate/adsorbent ion-molecular complexes of different ratios depending on the amount of cations, which are the main adsorptive active centers. For example, in MFI zeolite modification 5 form

of sodium cation, the formation of  $8\text{NH}_3:\text{Na}^+$  ion-molecular complex is formed by ammonia adsorption, and 24 hydrogen molecules form a wrap around sodium cation in the intersection of zeolite channels [12-14].

CaA (M-22), CaA (M-34),  $\text{Ca}_5\text{Na}_3\text{A}$  (MISS 624) and  $\text{Ca}_4\text{Na}_4\text{A}$  (Horst 50/50) zeolites are considered to be the main active centers of  $\text{Ca}^{2+}$  and  $\text{Na}^+$  cations in A-type zeolites. Also, since the amount of cations is in different proportions, adsorbate plays an important role in the amount of sorption of molecules. By determining the main thermodynamic characteristics of the adsorption of test molecules such as carbon dioxide, benzene, water and ammonia, the amount, nature and strength of energetically active centers in the same type of crystallographic positions are determined [15-24].

Step change of adsorption enthalpy of water and carbon IV oxide molecules in zeolites of type A (M-22, M-34, MISS-624, Horst 50/50) with alkaline earth and alkaline earth metal cations is the result of stoichiometric interaction with  $\text{Ca}^{2+}$  and  $\text{Na}^+$  cations. For example, water molecules in  $\text{CaA}_1$ ,  $\text{CaA}_2$  and  $\text{CaNaA}_1$  zeolites contain  $22\text{H}_2\text{O}/\text{e.c.}$ ,  $28\text{H}_2\text{O}/\text{e.c.}$  and  $30\text{H}_2\text{O}/\text{e.c.}$  respectively,  $7\text{CO}_2/\text{e.c.}$ ,  $6\text{CO}_2/\text{e.c.}$ ,  $8\text{CO}_2/\text{e.c.}$  and  $9\text{CO}_2/\text{e.c.}$  respectively, in  $\text{CaA}_1$ ,  $\text{CaA}_2$ ,  $\text{CaNaA}_1$  and  $\text{CaNaA}_2$  zeolites of carbon IV oxide molecules. A stepwise change in the formation of ion-molecular complexes has been determined [15-17, 24].

This article presents the results of adsorption isotherm, as well as the mechanism of adsorption, obtained by the method of adsorption-calorimetric experiment on synthetic zeolite of ammonia CaA (M-22).

**Methods.** The adsorption-calorimetric method used in this article allows obtaining the basic thermodynamic characteristics with high accuracy and revealing the detailed mechanisms of sorption processes in zeolite. The

experiment was carried out using a universal high-vacuum adsorption device. The device is adapted to measure the amount of adsorbate by both gas-volumetric and liquid-volumetric methods. A modified Tian-Calve type DAK-1-1A microcalorimeter was used as a microcalorimeter with high accuracy and stability.

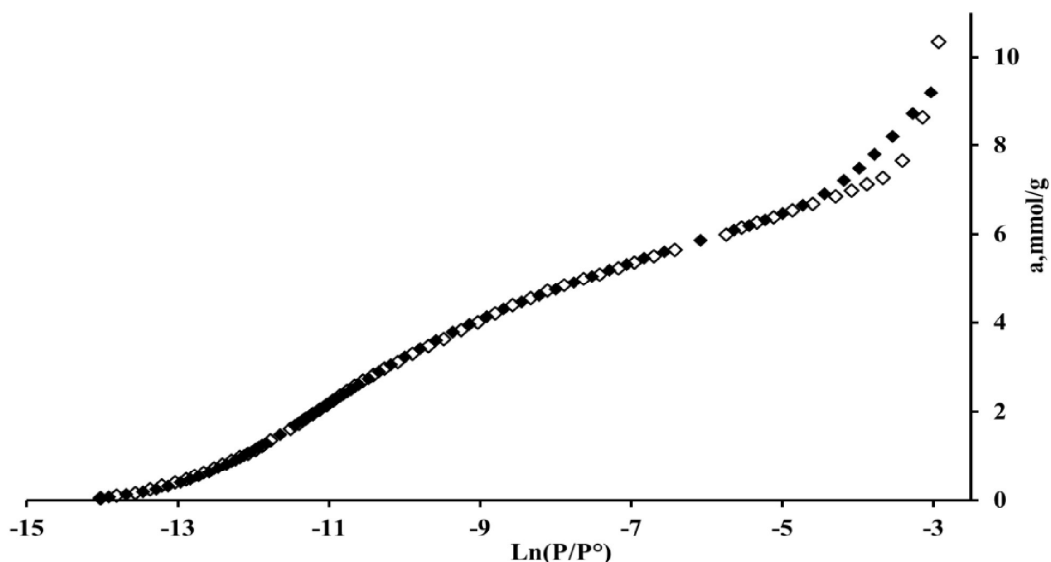
In the adsorption study, the adsorption of ammonia on CaA (M-22) zeolite at 303 K was studied and the adsorption isotherm was fully analyzed. The unit cell composition of this zeolite is  $\text{Ca}_{2,975}\text{Na}_{1,194}(\text{SiO}_2)_{12}(\text{AlO}_2)_{12}$ . Based on its chemical composition, the amount of calcium cations in 1 g of zeolite is 1.89 mmol/g and the amount of sodium cations is 0.76 mmol/g.

**Discussions.** The isotherm of ammonia adsorption on CaA (M-22) zeolite in logarithmic coordinates is presented in Fig. 1. At small saturations of the adsorption volume, the equilibrium relative pressure at the adsorption amount of 0.05 mmol/g is equal to  $P/P_s=8,11 \cdot 10^{-7}$  ( $R=0.00674$  torr). In this case,  $P_s=8750$  torr represents the saturation pressure of ammonia at a temperature of 303 K. Due to the high relative pressure of ammonia, the experiment was carried out up to 467 torr. The adsorption isotherm was brought to the adsorption amount of 10.35 mmol/g at a relative pressure of  $P/P_s=0.0534$  (or  $R=467$  torr).

The adsorption isotherm shows that cations in the zeolite matrix are in a strong ion-molecular complex bond with ammonia molecules in the initial region. The isotherm initially moves almost linearly towards the abscissa axis at the adsorption amount of  $\sim 0.76$  mmol/g up to  $P/P_s=4 \cdot 10^{-6}$  ( $R=0.04$  torr).

Based on the chemical composition of zeolite, the amount of sodium cations is equal to 0.76 mmol/g, that is, at the adsorption amount of  $\sim 0.8$  mmol/g, ammonia molecules form a  $1\text{NH}_3:\text{Na}^+$  monomer ion-molecular complex with

sodium cations in zeolite. The isotherm changes linearly with a partial tilt to  $P/P_s=1 \cdot 10^{-5}$  ( $R=0.088$  torr)  $\sim 1.52$  mmol/g adsorption of  $2\text{NH}_3:\text{Na}^+$  dimer, 2.3 mmol/g adsorption amount and  $P/P_s=1.76 \cdot 10^{-4}$  ( $R=0.154$  torr) at a relative pressure of  $3\text{NH}_3:\text{Na}^+$  trimer, 3 mmol/g adsorption amount and  $P/P_s=3.5 \cdot 10^{-4}$  ( $R=0.34$  torr) at a relative pressure of  $4\text{NH}_3:\text{Na}^+$  tetramer ion-molecular complex is formed.



**Figure 1. Adsorption isotherm of ammonia at 303 K on CaA (M-22) zeolite. In the  $\diamond$ -experiment,  $\blacklozenge$ -values of the general equation of the theory of volumetric saturation of micropores (VMOT)**

The subsequent adsorption isotherm graph of ammonia molecules changes regardless of the amount of sodium cations in the zeolite, which means that the adsorption process in the first coordination sphere with sodium cations of ammonia molecules is completed. This is also confirmed by the relative increase in the equilibrium pressure after the formation of the  $4\text{NH}_3:\text{Na}^+$  tetramer ion-molecular complex.

The regular change of the isotherm in accordance with the zeolite active centers corresponds to the amount of calcium cations, the second active center in its content,  $\sim 1.9$  mmol/g, that is, ammonia molecules begin to adsorb on  $\text{Ca}^{2+}$  cations. Value of relative pressure  $P/P_s=6 \cdot 10^{-4}$  ( $R=6$  torr) and 5 mmol/g adsorption of  $1\text{NH}_3:\text{Ca}^{2+}$  monomer, amount of adsorption 6.8 mmol/g and relative pressure  $P/P_s=0.0137$  ( $R=120$  torr)  $2\text{NH}_3:\text{Ca}^{2+}$  dimer forms ion-molecular complexes.

At the adsorption enthalpy obtained in the experiment, after the adsorption amount of  $\sim 7$  mmol/g, the enthalpy decreases to the heat of condensation of ammonia. But the change in differential enthalpy and entropy (above the entropy of liquid ammonia) changes in accordance with the amount of calcium cations in the zeolite and increases sharply, that is, ammonia molecules in the second coordination sphere of calcium cations adsorbate-adsorbate (ammonia-ammonia) with an adsorption amount of 8.7 mmol/g and  $P/P_s=0,05$  ( $R=380$  torr) at a relative pressure of  $\text{Ca}^{2+}:2\text{NH}_3:\text{NH}_3$  complex, the next 1.64 mmol/g ammonia molecules are also 10.52 mmol/g adsorption in the second coordination sphere and  $P/P_s=0,054$  ( $R=467$  torr) at a relative pressure of the sorption process ends by forming a  $\text{Ca}^{2+}:2\text{NH}_3:2\text{NH}_3$  complex.

Ammonia adsorption isotherm on CaA (M-22) zeolite is fully described using the three-state VMOT equation [25]:

$$a = 4.77 \exp[-(A/28.69)^7] + 2.015 \exp[-(A/17.63)^4] + 5.6 \exp[-(A/8.19)^3] \quad (1)$$

where,  $a$  is the adsorption value (mmol/g),  $A = RT \ln P_s/P$  is the free energy and represents the work (kJ/mol) done in transferring the gas to the equilibrium gas phase. From Figure 1, it can be seen that the calculated values in VMOT are in full agreement with the experimentally obtained adsorption amount up to 7 mmol/g. The first two terms of the equation represent the adsorption of ammonia molecules on the active centers of sodium and calcium cations of zeolite. The amount of adsorption in the formation of  $4\text{NH}_3:\text{Na}^+$  and  $1\text{NH}_3:\text{Ca}^{2+}$  is equal to 4.93 mmol/g, which corresponds to the value of  $a_{01} = 4.77$  mmol/g of equation 1. The amount of adsorption in the formation of  $4\text{NH}_3:\text{Na}^+$  and  $1\text{NH}_3:\text{Ca}^{2+}$  complexes is equal to 6.82 mmol/g, and the sum of the values of equation 1  $a_{01} = 4.77$  mmol/g and  $a_{02} = 2.015$  mmol/g fully corresponds to 6.92 mmol/g.

Since the adsorption process follows an exponential equation, the 3rd term of equation 1 has almost no effect from a small saturation pressure to a relative pressure of  $P/P_s$  ( $R = 120$  torr) and is weak. that is, it represents the sorption process

resulting from the mutual Waa-der-Waals interaction of the adsorbate molecule in the second coordination sphere.

**Discussions.** The isotherm of ammonia molecules in CaA (M-22) nanostructured zeolite was studied and the free energy values were calculated. Tetramer  $4\text{NH}_3:\text{Na}^+$  in the first coordination sphere with sodium cations in the  $S_I$  va  $S_{II}$  positions of zeolite, dimer  $2\text{NH}_3:\text{Ca}^{2+}$  ion-molecular complexes with calcium cations in the first coordination sphere, then two ammonia molecules with the initially adsorbed ammonia molecules in the second coordination sphere  $\text{Ca}^{2+}:2\text{NH}_3:2\text{NH}_3$  - was found to form molecular complexes. It was proved that the mechanism based on the coefficients of the adsorption amount obtained on the basis of the volume theory of micropores fully corresponds to the mechanism based on the values obtained in the experiment. It was found that 65% of the total adsorption amount up to 50 torr corresponds to the adsorption of sodium and calcium cations, the main active centers of zeolite.

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## RHEOLOGICAL PROPERTIES OF AMMOPHOSPHATE PULPS OBTAINED USING PHOSPHORITE POWDER OF THE KHODJAKUL DEPOSIT

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