Isotherms, Differential Heats Of Benzene Adsorption in zeolites LiLSX and NaLSX

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Abstract: *The study of molecules adsorption of different geometric and electronic structure by zeolites of different composition and structure is of great interest to study the effect on the adsorption of chemical nature of adsorbents surface. Zeolites are porous crystals, so the micropore structure of zeolites is determined with high accuracy, their adsorption properties are physical-chemical constants, which can be calculated theoretically based on the calculation of the potential interaction energy adsorbate-adsorbate and adsorbate-adsorbent in zeolites. These data are of great interest for the development of the theory of adsorption forces and intermolecular interactions. However, the calculation of the potential energy of adsorption is difficult due to the complexity of the crystal structure and the potential field in the zeolite channels. The high benzene content degrades the environmental and technological properties of fuels. To select a highly efficient sorbent for dearomatization of fuel, studies of regularities of benzene adsorption in various zeolites are necessary. The isotherm, differential heats of benzene adsorption in zeolites LiLSX and NaLSX at 303K were measured using the adsorption calorimetry method. The adsorption isotherm is quantitatively reproduced by VOM theory equations.*

Keywords: *Isotherm, differential heats, LiLSX zeolite, NaLSX zeolite, benzene, adsorption calorimetry*

1. INTRODUCTION

Zeolites are carcass-shaped aluminosilicate crystals composed of alkaline and alkaline earth metals, heated to constant temperature, aluminum and silicon oxides are not exposed to water and slowly evaporate, during which time carcasses are formed for the entry of small molecules consisting of regular channels and cavities [1]. Because synthetic zeolites have the same composition and structure, they are used for more industrial purposes than natural zeolite analogues [1].

In order to achieve an optimum performance for the adsorption column, it is of immense importance to understand the adsorbent–adsorbate interactions occurring, in terms of both the thermodynamics and kinetics of the processes. The dynamics of an adsorption column are influenced by several factors, including its geometry, operating scheme and conditions, the characteristics of mass transfer, the effects of adsorption heat and the type of adsorption equilibrium isotherm involved. In the present study, adsorption equilibrium isotherms for the pure gases and for a gaseous mixture were obtained for a lithium-exchanged low-silica (Si/Al = 1.0), X zeolite (LiLSX) and a carbon molecular sieve (CMS). Such isotherms were then employed to study the effect of competitive adsorption amongst the constituent gases and thereby establish their suitability for He purification. LiLSX is a synthetic crystalline aluminosilicate with a regular micropore structure. The zeolite possessed a faujasite-type structure (FAU) with a pore size of 9 Å [2, 3].

LSX zeolite is obtained by the cation exchange method to type X zeolites, and the cation exchange in these zeolites reaches a maximum. Further increase in the number of cations does not significantly affect the adsorption amounts. The results obtained are similar to the adsorption of type X zeolites [4, 5].

In the faujasite framework, four β-cages of sodalite are linked with four nearest sodalite cages through four D6R units. The sodalite cages are linked together to create the α-cages (also referred to as supercages). These supercages have an internal diameter of 1.3 nm, which is accessible through windows with a diameter of 0.74 nm composed by 12-membered-rings [6]. Sites I and I′ are located at the D6R faces linking the sodalite cages. Sites II and II′ are located at the open hexagonal (S6R) faces. Finally, sites III and III' are located at the surface of the supercage.

In this chapter the results of molecular simulation by means of Monte Carlo method were presented. This method allowed describing the adsorption of xylenes on NaLSX and BaLSX type zeolites. Adsorption in bulk-crystal phase and adsorption in a model taking into account the presence of a surface were investigated. The implementation of the force field was validated through the adsorption of mixture/pure xylenes in the bulk-crystal phase. The validation was conducted through the comparison with experimental data (either adsorption capacit, enthalpy or selectivity), observed both in this thesis and in the literature. Despite a simplified approach has been implemented in this work to minimize the energetic impact of the surface reconstruction (by minimizing the total dipole of the system), it is worth mentioning that the use of ab initio methodology would allow for a more accurate minimization of the surface energy. Nevertheless, the implemented methodology remains valid for the comprehension of the surface adsorption phenomena [7].

Adsorption of benzene molecules in type X and Y zeolites of fojasite was carried out by adsorption microcalorimetric method in a high-vacuum adsorption device and the complete thermodynamic properties of adsorption processes were described [8-23].

2. RESEARCH METHODS AND AREAS

For calorimetric study, LiLSX-type powdered synthetic zeolite ((Linda) Si / Al = 1) obtained using the ion exchange method was used. The composition of the elemental cells of zeolites obtained for the study is LiLSX - $Li_{96}Si_{96}Al_{96}O_{384}$ and is NaLSX - $Na_{96}Si_{96}Al_{96}O_{384}$Absolute

benzene was selected for adsorption. In the determination of LiLSX and NaLSX zeolites benzene adsorption was carried out in a high-vacuum adsorption calorimetric set [24,26].

The adsorption-calorimetric method used in this work allows one to obtain high-precision molar thermodynamic characteristics, as well as to reveal the detailed mechanism of adsorption processes occurring on adsorbents and catalysts. Adsorption measurements and dosage of adsorbate were carried out using a universal adsorption unit, in the working section of which exclusively mercury gates were used, replacing taps with lubrication [26]. The installation allows for dosage of the adsorbate by both gas-volumetric and volumetric-liquid methods. A modified DAK 1-1 calorimeter with high accuracy and reliability was used as a calorimeter.

3. RESULTS AND DISCUSSIONS

Benzene adsorption isotherms to LiLSX zeolite were determined by volumetric method at a temperature of 303 K. Figure 1 shows isotherm adsorption of benzene in LiLSX zeolite. The isotherm consists of a logarithmic (*ln*), adsorption (*N*) (*N*-supercage and 1/8 C_6H_6 molecule number in unit cell) and adsorption process over the entire pressure equilibrium range. Figure 1 shows the results of the experiment with the number 1 and the number 2 with the symbols processed using the equation of VMOT. At the initial saturation it is possible to observe a slight deflection of the isothermal marks, where the isotherm is ln (p/p°) = - 12, and the adsorption is up to $N = 1$ C_6H_6/1/8 u.c.. The isotherm index then appears as a vertical line until ln (p/p°) = - 8.35, after adsorption 4.76 C_6H_6/1/8 u.c. the isotherm bends and intersects towards the adsorption axis.
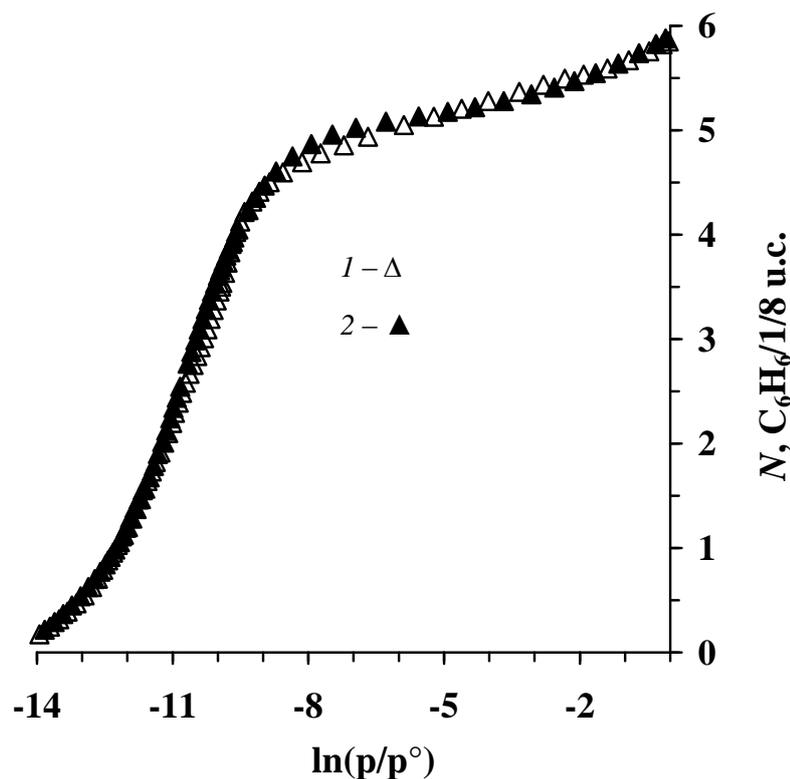
Figure 1. Benzene adsorption isotherm in the zeolite LiLSX at
303К.

1 - experimental data.

2-estimating data by VMOT.

Adsorption isotherms were described using the three-term equation of the VMOT [27,28]:
$N = 1,669\exp[-(A/32,27)^{10}]+3,257\exp[-(A/27,07)^{10}]+0,982\exp[-(A/9,06)^{1}]$ (LiLSX),
$N = 2,67\exp[-(A/32,07)^{10}]+1,79\exp[-(A/27,98)^{18}]+0,74\exp[-(A/10,21)^{1}]$,
(NaLSX)

N – adsorption in micropores C_6H_6/1/8 u.c., $A = RT \ln(P^o/P)$ - adsorption energy, kJ / mol.
Figure 2 shows the benzene adsorption isotherm to NaLSX zeolite. The isotherm (abscissa) axis consists of a logarithmic (ln), adsorption (N) (N-super-void and 1/8 C_6H_6 molecule number of elementary cell) (ordinal) axis, which allows us to imagine the adsorption process over the entire pressure equilibrium range. Figure 1 shows the results of the experiment with the number 1 and the number 2 with the characters redefined using the equation of the volume saturation theory of micropores. The relative pressure of the adsorption isotherm of the initial benzene molecules $7 \cdot 10^{-5}$ mm.of.mer., where the logarithmic value of the isotherm is ln (p / p °) = - 14.33. When a single molecule of benzene molecules initially fills the zeolite micropores, a slight bending of the isotherm line is observed, with the isotherm going from ln (p / p °) = - 14.33 to ln (p / p °) = - 12.8, while the adsorption N=1 C_6H_6/1/8 u.c. Then the isotherm index ln (p / p °) = - 10.54 until a vertical line appears, where adsorption 4.0 C_6H_6/1/8 u.c. and 3 molecules of benzene molecules are adsorbed. In the adsorption of the next 1.12 benzene molecule, the isothermal lines converge sharply toward the adsorption

axis, and when the isotherm logarithmic value reaches ln (p / p °) = - 0.22, the relative pressure is 96 mm.of.mer. and the saturation of benzene at a temperature of 30^0 C approaches the vapor pressure.
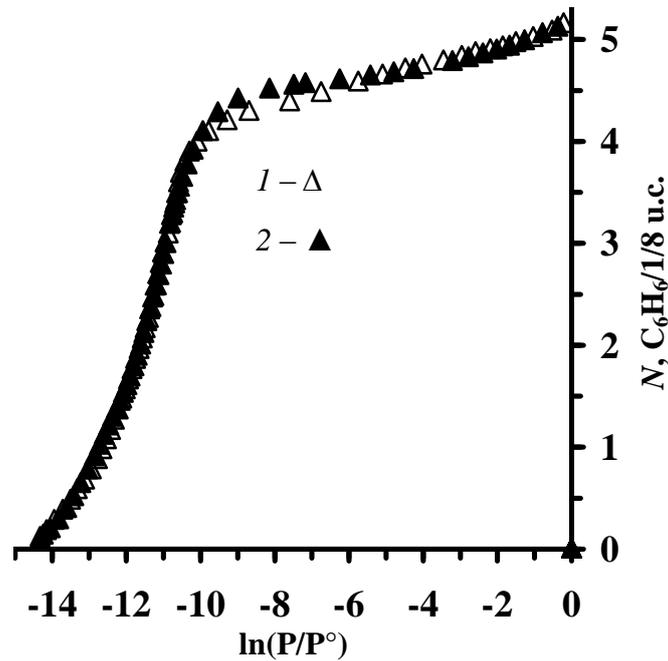

Figure 2. Benzene adsorption isotherm in the zeolite NaLSX at 303K.

1 - experimental data. 2-estimating data by VMOT.

The adsorption isotherms are known from the degree values of each term in the MHTN equation, i.e., the first and second limit levels are 10 and 18, indicating that these values extend to the zeolite micropores. The third limit is a small degree, in which benzene molecules go through the saturation phase and the adsorbate adsorbate molecules interact.
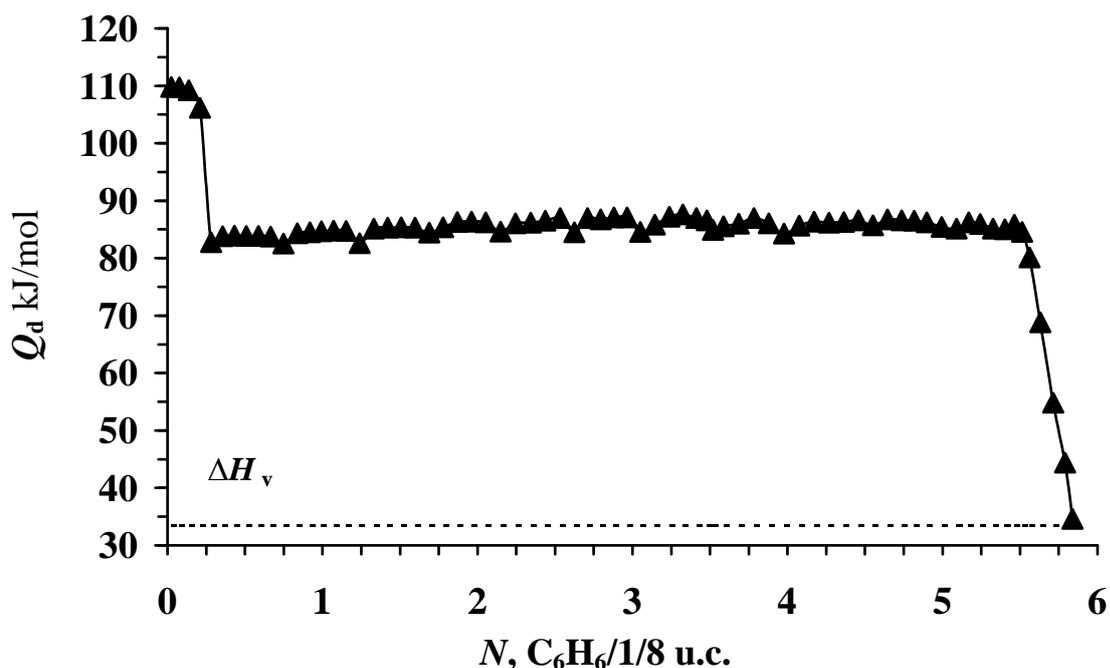
Figure 3 The differential heat of benzene adsorption in the zeolite LiLSX at
303K. The horizontal dashed line is heat benzene condensation at 303K.

Figure 3 shows the differential heat (Q_d) change in the amount of benzene adsorption (N) in LiLSX zeolite. Benzene adsorption is indicated by thermal condensation (Q_d) dashed lines. The stable differential heat of benzene adsorption is ~ 2.61 times higher than the thermal condensation. The change in the differential heat of adsorption has its own characteristics. The differential heat starts at 109.79 kJ / mol and decreases slowly to 106.17 kJ / mol, where the adsorption is in the range of 0.02-0.2 C6H6 / 1/8 u.c. The adsorption heat decreases from 106.17 kJ / mol to 82.76 kJ / mol and the adsorption reaches 0.28 C_6H_6/1/8 u.c. The differential heat at 82.76 kJ / mol has three maximums, and the adsorption intervals are 0.28-0.74 C_6H_6/1/8 u.c., 0.74-1.23 C_6H_6/1/8 u.c. and 1.23-1.68 C_6H_6/1/8 u.c. In addition, the adsorption heat goes to Q_d = 84.57 kJ / mol at N = 1.68 C_6H_6/1/8 u.c. And then to Q_d = 85.11 kJ / mol to the maximum value N = 5.09 C_6H_6/1/8 u.c. The interaction of adsorbate-adsorbate leads to an increase in the adsorption heat due to the contribution of the third and fourth benzene molecules to the total energy at the entrance of supercage. The heat form of fifth molecule is slight wave, and the adsorption is N = 5.46 C_6H_6/1/8 u.c. In the final stage, the temperature rises slightly and falls sharply at 303 K. Modeling the benzene / zeolite system shows that the fifth benzene molecule is located in a 12-ring oxygen window that separates the supercages from each other (position W).

The adsorption heat can be divided into the following parts according to the Q_d wave curve: 0.28-0.74, 0.74-1.23, 1.23-1.68, 1.68-2.14, 2.14-2.62, 2.62-3.05, 3.05-3.52, 3.52-3.97, 3.97-4.55, 4.55- 5.09 C_6H_6/1/8 u.c. The curve ends with segments in the range of 5.09-5.84 C_6H_6/1/8 u.c. LiLSX adsorbs 5.84 molecules of benzene of 1/8 u.c.

LiLSX zeolite contains Si / Al = 1: 1 ratio. The zeolites used in our previous studies have Si / Al> 1.09. According to the literature [29], the distribution of cations in the zeolite is as

follows: 4 cations in S_I position in the center of six- membered oxygen rings, 4 cations in S_{II} position near the eight-membered oxygen rings in the large cavity and the remaining 4 cations are located in $S_{III'}$ position There are 12 cations of 1/8 unit cell, or a total 96 cations of elementary cell. As can be seen from the content, zeolites have a very high density. Due to the size of the benzene molecule, it is not adsorbed in six-membered oxygen ring, which is approximately ~ 0.25 nm. Adsorption occurs mainly in supercages. The curve shows high adsorption values at $N = 0.28$ $C_6H_6/1/8$ u.c. in the $S_{III'}$ position. Because it is located near the entrance ring of the supercage, molecules are adsorbed with high energy ($Q_d = 109.79$ kJ / mol).

The second section at ~ 82.68 kJ / mol between 0.28–0.74 and 0.74–1.23 $C_6H_6/1/8$ u.c., which is explained by the adsorption on lithium cations located in the four-membered oxygen rings (S_{III}). The formation of cations in position S_{III} is associated with the migration of cations from position S_I. The migration of lithium cations is an active process in which the adsorption heat generated by the sorption of benzene would proceed at a much lower energy if the cations were located directly in the supercages. Lithium cations in position S_{III} have a preferred adsorption center than cations in position S_{II}. After benzene is adsorbed on Li^+ cations in position S_{III}, it begins to be adsorbed on Li^+ in position $S_{III'}$. In the next places, two sections of the lithium cations in positions $S_{III'}$ and S_{II} have ~ 0.475 $C_6H_6/1/8$ u.c. from (~ 0.475x2 = 0.95) benzene molecules are adsorbed (0.28-0.74, 0.74-1.23 $C_6H_6/1/8$ u.c.). Six sections: 1.23-1.68, 1.68-2.14, 2.14-2.62, 2.62-3.05, 3.05-3.52 and 3.52-3.97 $C_6H_6/1/8$ u.c. apply only to adsorption of lithium cations in the S_{II} position from 0.457 $C_6H_6/1/8$ u.c. The remaining sections each reflect adsorption in $S_{III'}$ and S_{II} from 0.58 $C_6H_6/1/8$ u.c. If we assume that each of these sections adsorb at S_{II} 0.457 $C_6H_6/1/8$ u.c. the rest corresponding to position $S_{III'}$ 0.808 $C_6H_6/1/8$ in five sections, the molecule is adsorbed (5 x 0.1616 = 0.808). In general, taking into account the second and third parts, in the S_{III} position 0.808 + 0.56 = 1.368 $C_6H_6/1/8$ u.c. adsorbed. As for the adsorption at the S_{II} position, according to the six sections, there are 2,742 $C_6H_6/1/8$ u.c. according to these centers. (6x0.457 = 2.742) and plus 1.12 $C_6H_6/1/8$ u.c. (2 to 0.56 $C_6H_6/1/8$ u.c.). 3,862 $C_6H_6/1/8$ u.c. adsorb in position S_{II}.

Figure 4 shows the differential heat according to the amount of benzene adsorption (N) on NaLSX zeolite (Q_d) given the graph of change. Benzene adsorption heat condensation ($\square\square_v$) indicated by ring lines. Benzene adsorption is stable differential heat from thermal condensation ~2,5 is equally high. The change in the differential heat of adsorption has its own characteristics. The differential heat starts at 107.06 kJ / mol and drops sharply to 82.5 kJ / mol, where the adsorption is in the range of 0.04-0.2 $C_6H_6/1/8$ u.c.. Adsorption heat for single molecule benzene adsorption up to 82.11 kJ / mol ($N=1$ $C_6H_6/1/8$ u.c.) will change. Adsorption after the maximum level of benzene sorption in the first and fourth molecules at a constant differential heat of 82.11 kJ / mol 1,0 $C_6H_6/1/8$ u.c. and 4,0 $C_6H_6/1/8$ u.c. represented by ea. In addition, the adsorption heat is in the form of full-length lines $Q_d=84.20$ kJ / mol $N=1.5$ $C_6H_6/1/8$ u.c., then the maximum value $N=2.5$ $C_6H_6/1/8$ u.c.. $Q_d=85$ kJ / mol. Adsorption 1.5 $C_6H_6/1/8$ u.c.. 4 $C_6H_6/1/8$ u.c.. The adsorbate-adsorbate interactions between the second and fourth lead to an increase in the adsorption heat due to the contribution of the second to fourth benzene molecules to the total energy in the space. Four C_6H_6/Na^+ the location of the complexes is in the form of a tetrahedron, and such clusters fill almost all the voids of the super voids. At the entrance of the fifth benzene molecule, the heat is in the form

of a wavy line and adsorption N=4.86 C_6H_6/1/8 u.c.. after passing the heat drops sharply. In the final stage, the temperature rises slightly and drops sharply depending on the temperature of the benzene condensation at 303 K. Modeling of the benzene / zeolite system shows that the fifth benzene molecule is located in a 12-ring oxygen window that separates the superpowers from each other. (W position). For zeolites with Na^+ cations in X and Y zeolites, this position has been discussed several times [30,31].
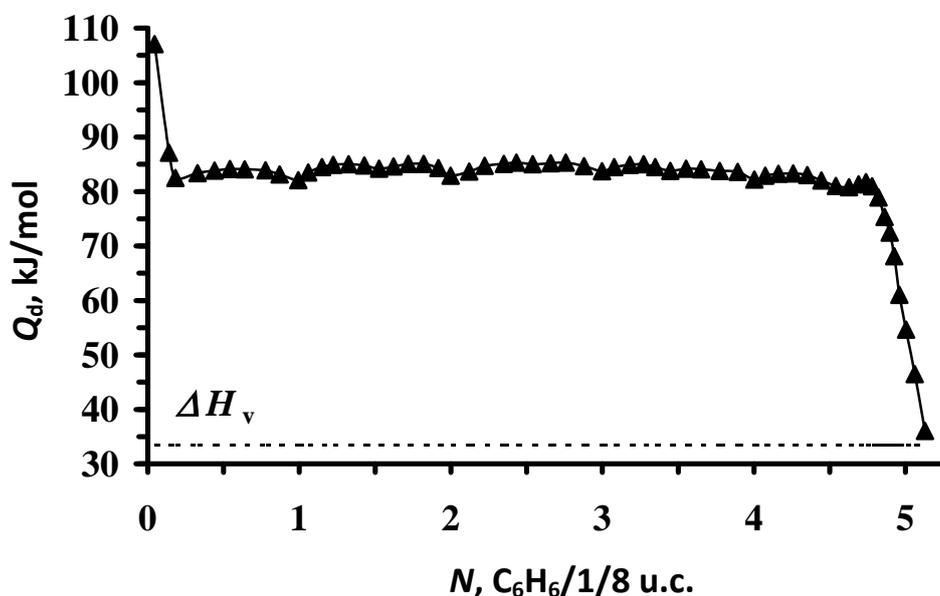

Figure 4 The differential heat of benzene adsorption in the zeolite
NaLSX at 303K. The horizontal dashed line is heat benzene
condensation at 303K.

Adsorption heat Q_d according to the wave curve can be divided into the following parts: 0.04-0.18, 0.18-0.99 (1-molecule), 0.99-1.52, 1.52-1.99 (2-molecule), 1.99-2.53, 2.53-2.99 (3-molecule), 2.99- 3.44, 3.44-4.0 (4-molecule), 4.0-4.54, 4.54-4.86 C_6H_6/1/8 u.c.. Curved line 4.86-5.12 C_6H_6/1/8 u.c. ends with segments in the range. NaLSX adsorbs a total of 5.12 molecules of benzene in 1/8 of zeolite or in one super void.

Zeolite NaLSX is a new faujasite type zeolite with Si/Al = 1. Until recently, the studied zeolites had Si / Al> 1.09. According to [29], the distribution of cations in the zeolite structure is as follows: 4 cations per 1/8 unit cell (1/8 u.c.) are located in the position $S_{I'}$ (at six-membered rings connecting cuboctahedra and hexagonal prisms), 4 cations per 1/8 u.c. are in position S_{II} (in six-membered rings connecting cuboctahedra and large cavities) and the remaining 4 cations at 1/8 u.c. are in position $S_{III'}$ (in four-membered rings of a large cavity). In total, there are 12 cations per 1/8 u.c., Or the super cavity, or 96 cations per unit cell. As seen from the composition, zeolites have a very high charge density.

As can be seen from the content, zeolites have a very high density. Due to the size of the benzene molecule, it is not adsorbed on six-membered oxygen glass, i.e., sodalite cavities, which is approximately ~ 0.25 nm. Adsorption occurs mainly in super voids. Initially, the high adsorption temperatures are due to the adsorption of benzene on Na^+ cations in the S_{III}

'position located in the superpowers. ($N = 0,18$ C_6H_6/1/8 u.c.) related to. Because it is located near the entrance window to the supercosm, the adsorbate molecules are initially adsorbed into this cavity and therefore travel with high energy (Q_d=107,06 kJ / mol).

The second section 0.18-0.99 and 0.99-1.52 C_6H_6/1/8 u.c.. The temperature in the range is ~ 83.68 kJ / mol, in these four-membered oxygen rings (S_{III}) explained by adsorption on sodium cations. S_{III} this is the appearance of cations in the position $S_{I'}$ associated with the migration (migration) of cations in the position. The migration of sodium cations is an active process, and if the cations were located directly in the superclimates, the adsorption heat generated by the sorption of benzene would proceed at a much lower energy. S_{III} sodium cations in position S_{II} the adsorption center is preferred over the cations in the position because the 4-membered oxygen ring is located in the part slightly bent into the supercollective. Because the size of the Na^+ cations is small, the SII is more submerged in the 6-membered oxygen ring of the position. Also, benzene molecules adsorbed on sodium cations in the SII position, cations forming the 6-membered oxygen ring, and negatively charged oxygen atoms do not move more easily.. $S_{III'}$ after benzene adsorption on Na^+ cations in position, S_{III} in position Na^+ begins to be adsorbed. S_{III} and S_{II} to sodium cations in the cavities ~0.5 C_6H_6/1/8 u.c.. (~0.5x2=1) benzene molecules are adsorbed on the mixture (0.04-0.5, 0.5-1.0 C_6H_6/1/8 u.c.). Seven sections: 1.0-1.5, 1.5-2.0, 2.0-2.5, 2.5-3.0, 3.0-3.5, 3.5-4.0 and 4.0-4.5 C_6H_6/1/8 u.c.. only S_{II} to sodium cations in position 0.5 C_6H_6/1/8 u.c.. suitable for adsorption. In these sections, the adsorption takes place while maintaining the duration of the heat value and is ~ 83.70 kJ / mol. In the remaining sections 0.62 C_6H_6/1/8 u.c. S_{III} and S_{II} reflects adsorption. As the adsorption heat goes with the high and low energy, it goes in a very small part of the adsorption. If we have each of these sections S_{II} and adsorption 0,5 C_6H_6/1/8 u.c.. Given the stain, S_{III} the residual corresponding to the position is 0.18. In five sections 0.5 C_6H_6/1/8 u.c.. the molecule is adsorbed (5 x 0.18 =0.8). In general, in the S_{III} position, taking into account the second and third parts 0.8+0.62=1.42 C_6H_6/1/8 u.c. adsorbed. As for the adsorption on the S_{II} position, according to the seven sections, according to these centers 3.5 C_6H_6/1/8 u.c.. (7x0.5=3,5) and plus 1.12 C_6H_6/1/8 u.c. (2 - 0,56 C_6H_6/1/8 u.c..) adsorbed. Total S_{II} 3,5 C_6H_6/1/8 u.c.., S_{III} 1.42 C_6H_6/1/8 u.c.. and 0.12 molecules of benzene are adsorbed on the adsorbate adsorbate. The 0.12 benzene molecule binds the sodium cations in the S_{III} and S_{II} cavities to each other. This causes the sodium cations in the S_I cavities to migrate to the S_{III} cavity.

4. CONCLUSION

0.788 C_6H_6/1/8 u.c. adsorbed in position $S_{III'}$, 3,862 C_6H_6/1/8 u.c. in position S_{II} and 1,368 C_6H_6/1/8 u.c. in position S_{III}. Of the 12 lithium cations in 1/8 of the zeolite matrix, 5,838 are involved in the adsorption process. In the last section, after adsorption of 5.46 C_6H_6/1/8 u.c. near the end of the adsorption process, the heat begins to decrease from 85.84 to 80.15 kJ / mol as usual. At the end of the process, the adsorbed molecules are adsorbed more densely and the 0.2 C_6H_6/1/8 or benzene molecules are redistributed. Their adsorption heat rises sharply to the level of 8,621 kJ / mol. The redistribution of benzene molecules begins mainly after adsorption of 5.56 C_6H_6/1/8 u.c.

NaLSX zeolite the adsorption of benzene vapor on zeolite was described using the equation of the volumetric saturation theory of micropores. To saturate the voids of NaLSX zeolite

with benzene, 0.2 $C_6H_6/1/8$ u.c. $S_{III'}$ cations in position S_{II}, cations in position S_{II} 3,5 and cations in position S_{III}. 1,42 $C_6H_6/1/8$ u.c.. adsorbed. Of the 12 sodium cations in 1/8 of the zeolite matrix, 5.12 are involved in the adsorption process. At the end of the process, the adsorbed molecules are adsorbed more densely and 0.2 $C_6H_6/1/8$ u.c. benzene molecules are redistributed. The redistribution of benzene molecules is mainly 4,98 $C_6H_6/1/8$ u.c.. begins after adsorption.

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