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# Thermodynamics of benzene adsorption in NaLSX zeolite

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**ABSTRACT:** Differential heats, isotherm, molar entropy and thermokinetics of benzene vapor adsorption in a zeolite NaLSX have been measured by Tian-Calvet-type microcalorimeter and volumetric system at 303 K. The average molar integral entropy of benzene adsorption in zeolite NaLSX is  $-73,68 \text{ J/mol}\cdot\text{K}$ , which indicates the localization of benzene molecules in NaLSX zeolite. The adsorption isotherm is quantitatively reproduced by VOM theory equations.

**KEYWORDS:** Isotherm, differential heats, differential entropies, thermokinetics NaLSX zeolite, benzene, adsorption calorimetry

## I. INTRODUCTION

In the faujasite framework, four  $\beta$ -cages of sodalite are linked with four nearest sodalite cages through four D6R units. The sodalite cages are linked together to create the  $\alpha$ -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 [1]. 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 capacity, 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 [2].

Adsorption of benzene molecules in type X and Y zeolites of faujasite was carried out by adsorption microcalorimetric method in a high-vacuum adsorption device and the complete thermodynamic properties of adsorption processes were described [3-18].

**Methods and materials.** 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 [19]. 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.

**Results and discussion.** NaLSX benzene adsorption isotherms to zeolite were carried out by volumetric method at a temperature of 303 K. Figure 1 shows the benzene adsorption isotherm to NaLSX zeolite. The isotherm (abscissa) axis consists of a logarithmic ( $\ln$ ), adsorption (N) (N-super-void and  $1/8 \text{ C}_6\text{H}_6$  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  $7 \cdot 10^{-5} \text{ mm.of.mer.}$ , where the logarithmic value of the isotherm is  $\ln(p/p^\circ) = -14.33$ . 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  $\ln(p/p^\circ) = -14.33$  to  $\ln(p/p^\circ) = -12.8$ , while the adsorption  $N=1 \text{ C}_6\text{H}_6/1/8 \text{ u.c.}$  Then the isotherm index  $\ln(p/p^\circ) = -10.54$  until a vertical line appears, where adsorption  $4.0 \text{ C}_6\text{H}_6/1/8 \text{ u.c.}$  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  $\ln(p/p^\circ) = -0.22$ , the relative pressure is 96 mm.of.mer. and the saturation of benzene at a temperature of 30<sup>0</sup> C approaches the vapor pressure.

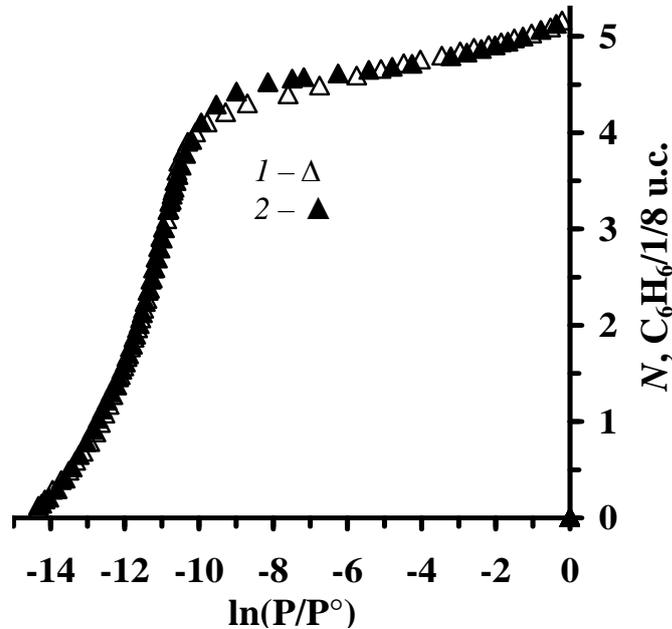


Figure 1. Benzene adsorption isotherm in the zeolite NaLSX at 303K.

1 - experimental data.

2-estimating data by VMOT.

Adsorption isotherm of ammonia in the NaLSX zeolites is satisfactorily described by three-term equation of the theory of volumetric micropore occupancy (VMOT) [20, 21]:

$$N = 2,67\exp[-(A/32,07)^{10}] + 1,79\exp[-(A/27,98)^{18}] + 0,74\exp[-(A/10,21)^1],$$

$N$ – adsorption in micropores  $C_6H_6/1/8$  u.c.,  $A=RT \ln(P^\circ/P)$  – adsorption energy, kJ/mol.

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 2 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 ( $\square\square,$ ) indicated by ring lines. Benzene adsorption is stable differential heat from thermal condensation  $\sim 2,5$  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 [22,23].

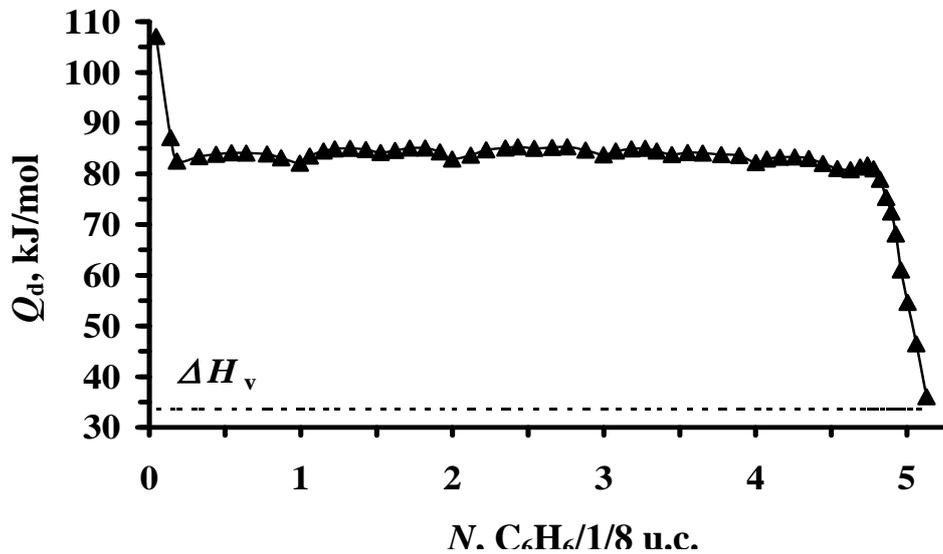


Figure 2 The differential heat of benzene adsorption in the zeolite NaLSX at 303K.

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 [24], 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  $S_{II}$  is more submerged in the 6-membered oxygen ring of the position. Also, benzene molecules adsorbed on sodium cations in the  $S_{II}$  position, cations forming the 6-membered oxygen ring, and negatively charged oxygen atoms do not move more easily..  $S_{III}$  after



benzene adsorption on  $\text{Na}^+$  cations in position,  $S_{\text{III}}$  in position  $\text{Na}^+$  begins to be adsorbed.  $S_{\text{III}}$  and  $S_{\text{II}}$  to sodium cations in the cavities  $\sim 0.5 \text{ C}_6\text{H}_6/1/8 \text{ u.c.}$  ( $\sim 0.5 \times 2 = 1$ ) benzene molecules are adsorbed on the mixture (0.04-0.5, 0.5-1.0  $\text{C}_6\text{H}_6/1/8 \text{ u.c.}$ ). 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  $\text{C}_6\text{H}_6/1/8 \text{ u.c.}$  only  $S_{\text{II}}$  to sodium cations in position 0.5  $\text{C}_6\text{H}_6/1/8 \text{ u.c.}$  suitable for adsorption. In these sections, the adsorption takes place while maintaining the duration of the heat value and is  $\sim 83.70 \text{ kJ / mol}$ . In the remaining sections 0.62  $\text{C}_6\text{H}_6/1/8 \text{ u.c.}$   $S_{\text{III}}$  and  $S_{\text{II}}$  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  $S_{\text{II}}$  and adsorption 0.5  $\text{C}_6\text{H}_6/1/8 \text{ u.c.}$  Given the stain,  $S_{\text{III}}$  the residual corresponding to the position is 0.18. In five sections 0.5  $\text{C}_6\text{H}_6/1/8 \text{ u.c.}$  the molecule is adsorbed ( $5 \times 0.18 = 0.8$ ). In general, in the  $S_{\text{III}}$  position, taking into account the second and third parts  $0.8 + 0.62 = 1.42 \text{ C}_6\text{H}_6/1/8 \text{ u.c.}$  adsorbed. As for the adsorption on the  $S_{\text{II}}$  position, according to the seven sections, according to these centers 3.5  $\text{C}_6\text{H}_6/1/8 \text{ u.c.}$  ( $7 \times 0.5 = 3.5$ ) and plus 1.12  $\text{C}_6\text{H}_6/1/8 \text{ u.c.}$  ( $2 - 0.56 \text{ C}_6\text{H}_6/1/8 \text{ u.c.}$ ) adsorbed. Total  $S_{\text{II}}$  3.5  $\text{C}_6\text{H}_6/1/8 \text{ u.c.}$ ,  $S_{\text{III}}$  1.42  $\text{C}_6\text{H}_6/1/8 \text{ u.c.}$  and 0.12 molecules of benzene are adsorbed on the adsorbate adsorbate. The 0.12 benzene molecule binds the sodium cations in the  $S_{\text{III}}$  and  $S_{\text{II}}$  cavities to each other. This causes the sodium cations in the  $S_{\text{I}}$  cavities to migrate to the  $S_{\text{III}}$  cavity.

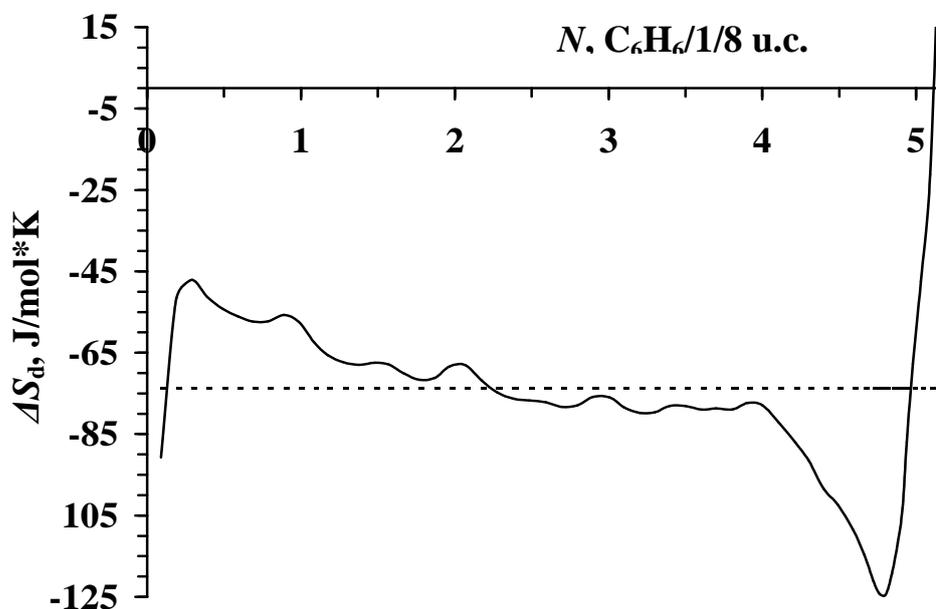
Figure 3 shows the differential entropy of benzene adsorption on NaLSX zeolite. The formula of the Gibbs-Helmholtz equation was used to calculate the differential entropy using the differential heat and isotherm values of benzene adsorption on NaLSX zeolite [25].

$$\Delta S_d = \frac{\Delta H - \Delta G}{T} = \frac{-(Q_d - \lambda) + A}{T}$$

$\lambda$ - thermal condensation,  $\Delta H$  and  $\Delta G$ - enthalpy and free energy change,  $T$  - temperature,  $Q_d$ - average differential heat.

The adsorption entropy obtained on the basis of the results of the study indicates the position of the adsorbent cavities, the adsorbate molecules in the cations on the surface. The large number of voids in the zeolite micropores at the beginning of the process and the high adsorption heat for the distribution of the benzene molecules to the pores indicate that the benzene molecules at the initial saturation are not strongly excited in the zeolite micropores.

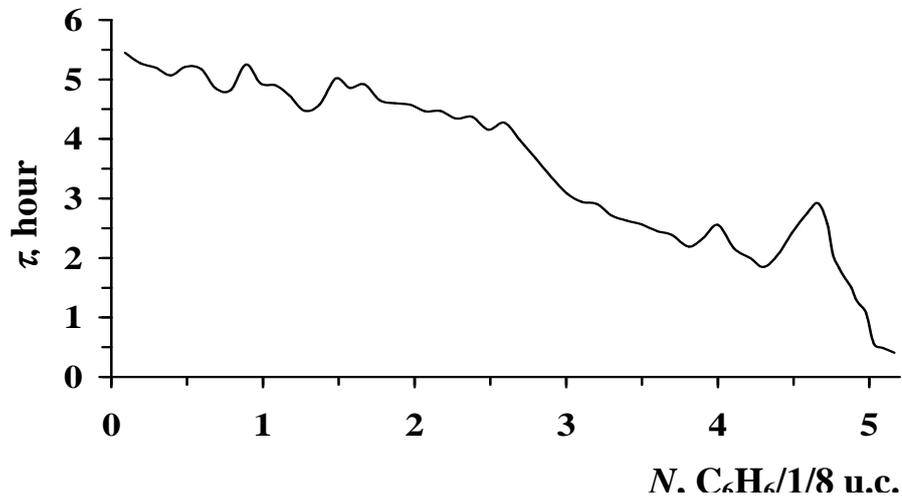
The differential entropy of adsorption initially starts at  $-90.7 \text{ J / mol} \cdot \text{K}$ , and the adsorption  $N = 0,094 \text{ C}_6\text{H}_6/1/8 \text{ u.c.}$  forms. Then the entropy rises and rises slowly to  $-47.4 \text{ J / mol} \cdot \text{K}$ , the adsorption entropy lines decrease in a stepwise manner, the adsorption 4  $\text{C}_6\text{H}_6/1/8 \text{ u.c.}$  when  $-72,9 \text{ J / mol} \cdot \text{K}$  and then a sharp decrease to  $-124.47 \text{ J / mol} \cdot \text{K}$  is observed.



**Figure 3.** The differential entropy of the benzene adsorption in the zeolite NaLSX at 303K. Entropy of liquid benzene is taken as zero. The horizontal dashed line – mean molar integral entropy.

Initially, due to the large number of voids in the zeolite, the cations are not fully saturated with benzene vapors, the adsorption entropy values are high and the adsorbate molecules are in an inert state. In the adsorption of 0.3 molecules of benzene vapors, it is adsorbed on cations close to the entrance glass in the cavity  $S_{III}$ . Adsorption  $N=0,31$   $C_6H_6/1/8$  u.c.. then gradually the differential entropy of adsorption in the form of a wavy line decreases  $N\sim 2,25$   $C_6H_6/1/8$  u.c.. below the mean integral entropy line. Adsorption of benzene molecules in the  $S_{II}$  supercapsid  $N=0,2$   $C_6H_6/1/8$  u.c.  $N=4,8$   $C_6H_6/1/8$  u.c.. goes in the range of up to.  $N\sim 5,03$   $C_6H_6/1/8$  u.c. benzene is very strongly sorbed in the zeolite matrix and the solid state at  $25^0S$  is close to the entropy value. Then adsorption  $N\sim 5,06$   $C_6H_6/1/8$  u.c.. then approaches the value of 0 from the mean integral lines. This results in the formation of complex compounds with  $Na^+$  cations that migrate from the  $S_{III}$  cavity inside the  $S_{II}$  cavity of the zeolite matrix while forming small wavy line views. Due to the large number of cations in these cavities, the energy distribution in the migration and adsorption of cations is orderly and strongly adsorbed. The average integral entropy is  $-73.68$  J / mol \* K.

Figure 4 shows the equilibrium time of benzene adsorption on NaLSX zeolite. In this zeolite, the equilibrium time lines are wavy. The equilibrium time of benzene adsorption to NaLSX zeolite is initially 5.5 hours. In LiLSX zeolite, benzene adsorption initially lasts 12 hours [13,18].



**Figure 4.**– The set-time of the adsorption equilibrium, depending on the size of the adsorption of benzene in the zeolite NaLSX at 303K

It takes more time for zeolite to break down into pores and due to the large number of cations located in the pores and the small amount of benzene molecules delivered. One benzene molecule (1 C<sub>6</sub>H<sub>6</sub>/1/8 u.c.) the heat equilibrium time in adsorption decreases to 5 h. 1.25 C<sub>6</sub>H<sub>6</sub>/1/8 u.c.. The equilibrium time in the adsorption of the benzene molecule is reduced to 4.5 hours, 1.5 C<sub>6</sub>H<sub>6</sub>/1/8 u.c.. rises to another 5 hours and 2 C<sub>6</sub>H<sub>6</sub>/1/8 u.c.. will be another 4.5 hours. 2.5 C<sub>6</sub>H<sub>6</sub>/1/8 u.c.. The heat balance time is reduced to 4 hours. 3.0 C<sub>6</sub>H<sub>6</sub>/1/8 u.c.. The equilibrium lasts for 3 hours. Adsorption amount of 2.5 hours in benzene adsorption of the fourth molecule 5 C<sub>6</sub>H<sub>6</sub>/1/8 u.c.. after which the equilibrium time gradually decreases to 30 minutes. At the end of the process, the equilibrium time is reduced to 10-20 minutes.

## II. CONCLUSION

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<sub>6</sub>H<sub>6</sub>/1/8 u.c. S<sub>III</sub> cations in position S<sub>II</sub>, cations in position S<sub>II</sub> 3,5 and cations in position S<sub>III</sub>. 1,42 C<sub>6</sub>H<sub>6</sub>/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<sub>6</sub>H<sub>6</sub>/1/8 u.c. benzene molecules are redistributed. The redistribution of benzene molecules is mainly 4,98 C<sub>6</sub>H<sub>6</sub>/1/8 u.c.. begins after adsorption. The average integral differential entropy is - 73.68 J / mol \* K, and the benzene molecules are adsorbed in the zeolite matrix without solid agitation. In the initial saturation, it takes a long time for the adsorption heat equilibrium to be established. Gradually, as the saturation increases, the adsorption thermokinetics decreases for a few minutes.

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