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Adsorption equilibria of binary ethylmercaptan/hydrocarbon mixtures on a NaX zeolite

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Abstract We present here coadsorption data of ethylmercaptan with *n*-heptane or toluene on NaX at 298 K in the aim to use this adsorbent for removing mercaptans from natural gas. Results show that NaX has an adsorption affinity for sulphur compound strong enough to perform a deep desulphurization. NaX adsorbs preferentially ethylmercaptan over a large domain of pore filling. A displacement of *n*-heptane by ethylmercaptan is even observed. However, toluene becomes preferentially adsorbed at high filling.

The dependence of filling and composition on selectivities and failed prediction of coadsorption equilibria by the IAS Theory indicate that the adsorbed mixture behaves as a non-ideal solution. Calorimetric measurements of coadsorption heats show that the coadsorption process is not governed by enthalpic effects but by entropic effects consecutive to steric hindrance of molecules in confined spaces.

Keywords Coadsorption · Ethylmercaptan · *n*-heptane · Toluene · NaX · Calorimetry

1 Introduction

Natural gases contain some impurities as water, carbon dioxide, hydrogen sulphide and light mercaptans (mainly methyl- and ethylmercaptans), which must be removed before industrial use. Indeed, the environmental legislation will impose in a near future a drastic reduction of sulphur emission in the atmosphere (less than 20 molar ppm). On the other hand, natural gas used in fuel cell must be highly purified because sulphur acts as a fearful poison for catalysts that are used for conversion of methane into hydrogen. In the conventional process, the natural gas is firstly cleaned of the acid gases carbon dioxide and hydrogen sulfide in an amine-washing unit then dried in a glycol unit. However, these treatments do not eliminate completely sulphur compounds because light mercaptans are not acidic enough to be removed by this way. An additional step in the treatment process is required to reduce to a lower level the sulphur content in the gas. Gas purification by selective adsorption on zeolites is an alternative way that is in progress. Although papers on desulphurization of liquid fuels by adsorption on zeolites are more and more numerous these last six years (Yang et al. 2001; Velu et al. 2003; Hernandez-Maldonado and Yang 2003, Hernandez-Maldonado et al. 2004), fundamental studies on the adsorption of mercaptans in these nanoporous materials are rather scarce. As shown in previous papers (Weber et al. 2005; Benoit et al. 2005) the NaX zeolite, which has the advantage of being low cost shows a strong adsorption affinity and a large adsorption capacity for polar molecules and therefore, could be a good candidate for reducing mercaptan contents down to very low concentration. However, natural gases contain other impurities as C2-C7 alkanes and aromatic compounds (benzene, toluene…) that could be competitively adsorbed by the NaX zeolite and consequently could change the retention of mercaptans. Therefore, it was of great interest to study the coadsorption of ethylmercaptan with *n*-heptane or toluene, which are representative of impurities present in natural gas after conventional treatment. In the first part of this paper, single

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component adsorption data on the NaX zeolite are briefly recalled then, some predictions of adsorption selectivity of mercaptan with respect to hydrocarbon are attempted. The second part of the paper is dedicated to the presentation of adsorption equilibria of binary ethylmercaptan/hydrocarbon mixtures (coadsorption isotherms, coadsorption enthalpies and adsorption selectivities) on the NaX zeolite measured in a large range of gas mixture composition.

2 Experimental

The commercial NaX zeolite with silicon to aluminum ratio of 1.23 was supplied without binder by Union Carbide Corporation. The chemicals ethylmercaptan (ESH), *n*-heptane (HEP) and toluene (TOL) of purity better than 99% were provided by Sigma Aldrich. They were dried over a hydrophilic 4A zeolite before use. Adsorption of single components was studied by thermogravimetry under controlled vapor pressure with a McBain thermobalance constructed in house. About 15 mg of sample were exposed to gaseous adsorptive in the relative pressure range of 10^{-6} to 10^{-1} to obtain adsorption-desorption isotherms at 298 K. Isotherms were drawn step by step using a static method. Once a plateau of mass is recorded a following equilibrium is reached by increasing or decreasing the relative pressure. The experimental accuracies were ±0.01 mg for the mass, 1 K for the temperature and 1% for the relative pressure. Adsorption of binary ESH + HEP and ESH + TOL mixtures was studied by mean of a home made manometric apparatus coupled with a calorimeter, a gas phase chromatograph and a quadrupole mass spectrometer. A bypass system with a sample loop connected to a vacuum line was used to collect small amounts of gas mixture, which composition was determined according to pressure either by chromatography ($p > 0.1$ hPa) or by mass spectrometry ($p < 0.1$ hPa). Coadsorption isotherms were determined by exposing the sample to small amounts of gas mixture. Direct measurements of total pressure and composition of the gas before and after adsorption allowed to calculate the amount of each component adsorbed in the zeolite and therefore, their mole fraction in the adsorbed phase and the adsorption selectivity.

The advantage of the technique is that total and partial adsorption isotherms N^a or $N_i^a = f(p)_T$, coadsorption differential enthalpy curves $\Delta H^a = f(N^a)_T$ and, adsorption selectivity curves $\alpha_{\mathrm{ESH/HC}} = f(N^a)_T$ are simultaneously measured.

Coadsorption equilibria were determined at 298 K on a sample weight of about 500 mg and for pressures ranging from 10^{-4} to 14 hPa. The relative error on the pressure and the mole fractions of gas phase species was around 1%. The accuracy was about 0.15 molec·α^{-1} and 5 kJ·mol^{-1} for the adsorbed amount and the adsorption heat, respectively. The mole fractions of ethylmercaptan in the initial gas mixture, y^0_{ESH}, were 0.1, 0.25, 0.50, 0.75 and 0.90. Prior to each adsorption, the sample was out-gassed at 673 K under vacuum overnight. More details about the experimental conditions and the procedure can be found in previous papers (Weber et al. 2005, 2008).

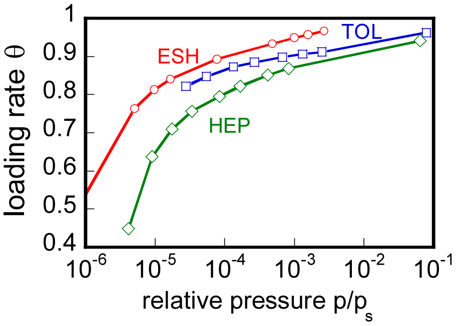

Fig. 1 Adsorption isotherms of single components ESH, HEP and TOL on the NaX zeolite determined by thermogravimetry at 298 K

3 Results and discussion

3.1 Adsorption of single components and prediction of selectivity

Adsorption isotherms of single components on the NaX zeolite obtained by thermogravimetry under controlled vapor pressure are given in Fig. 1. The loading rate ($\theta = \frac{V^a}{V^a_{\max i}}$) instead of N^a and the relative pressure ($\frac{p}{p_s}$) instead of pressure are used as coordinates to better compare the adsorbate/adsorbent systems. V^a and $V^a_{\max i}$ are the volumes adsorbed at p and p_s, respectively. They are calculated assuming that the density of the adsorbate is equal to the bulk liquid density. For the three adsorptives, the adsorption-desorption process is reversible. The maximal adsorption capacities given in Table 1 were determined by the Dubinin-Radushkevich model, assuming the density of the adsorbed phase is that of the liquid at 298 K. They never exceed the crystallographic volume of supercages of the NaX zeolite ($V^\alpha = 0.294$ cm^3·g^{-1}). Indeed, the adsorptive molecules are too large to enter sodalite cages. If ethylmercaptan entirely probes supercages, the other two adsorptives probe them only up to around 90% because of their larger molecular volumes (146.7 cm^3·mol^{-1} for HEP, 106 cm^3·mol^{-1} for TOL and 74.6 cm^3·mol^{-1} for ESH). Then, toluene and *n*-heptane appear to be more sensitive to confinement effects than ethylmercaptan. So, it is expected that the steric hindrance in supercages will influence the competitive adsorption of ethylmercaptan with *n*-heptane or toluene. From an energetic standpoint, the NaX zeolite exhibits a strong adsorption affinity for the three adsorptives as indicated by

Table 1 Adsorption capacities of the NaX zeolite for single components ESH, HEP and TOL and the corresponding differential molar enthalpies, entropies and Gibbs energies of adsorption extrapolated at zero filling at 298K

Component	$V^a_{\max i}$ ($\text{cm}^3 \cdot \text{g}^{-1}$)	ΔH^{a*}_{∞} ($\text{kJ} \cdot \text{mol}^{-1}$)	ΔS^{a*}_{∞} ($\text{J} \cdot \text{K}^{-1} \cdot \text{mol}^{-1}$)	ΔG^{a*}_{∞} ($\text{kJ} \cdot \text{mol}^{-1}$)
ESH	0.294	−65	−56	−48
HEP	0.270	−60	−108	−28
TOL	0.266	−66	−120	−30

the high loading rates reached at very low relative pressure. Adsorption affinity is so strong that Henry's law is never observed. Such an adsorption affinity is particularly interesting to perform a deep desulphurization of natural gas. Calorimetric differential molar adsorption enthalpies extrapolated at zero filling, which account for adsorbate-adsorbent interactions, are reported in Table 1. ΔH^{a*}_{∞} increases along the series TOL ~ ESH < HEP. Adsorbate-adsorbent interactions are of the same order of magnitude for ESH (sulphur atom-sodium cation specific interactions) and TOL (hydrogen-oxygen framework and π-electrons-sodium cation specific interactions). As expected, adsorbate-adsorbent interactions are slightly weaker for HEP than for ESH and TOL because n-heptane undergoes only non-specific interactions. Adsorption Gibbs energies at zero filling are calculated from adsorption enthalpies and isosteric adsorption entropies determined by calorimetric and thermogravimetric measurements (Table 1). ΔG^{a*}_{∞} increases along the sequence ESH < TOL < HEP indicating that the NaX zeolite exhibits the strongest adsorption affinity for ethylmercaptan. This result is in agreement with the shape of isotherms at low pressure. The fact that enthalpies and Gibbs energies do not vary in the same way, means that the entropic term plays an important part in the adsorption affinity. From Gibbs energies obtained for the adsorption of single components, it can be thought on the assumption of ideal binary mixtures that the NaX zeolite would preferentially adsorb ethylmercaptan. Moreover, $\alpha_{\text{ESH/TOL}}$ should be lower than $\alpha_{\text{ESH/HEP}}$. However, this is a rough estimation of selectivity. A more accurate prediction can be done by using the well-known Ideal Adsorption Solution Theory (Myers and Prausnitz 1965). Figure 2 shows the variation of the mole fraction of ESH at equilibrium in the gas phase with respect to that one in the adsorbed phase. IAST prediction agrees with the qualitative conclusions given by comparing the Gibbs energies. Ethylmercaptan should be favorably adsorbed by the NaX zeolite and the selectivity should be lower for toluene than for n-heptane.

3.2 Adsorption of binary mixtures

Total adsorption isotherms of binary mixtures have classical type I shape (Fig. 3). Whatever the composition of mixture, the amount adsorbed at the plateau of the isotherm lies between those of single components. Partial adsorption isotherms are continuously increasing with pressure for ESH + TOL mixtures. The higher the mole fraction of toluene in the initial mixture, the higher is the amount of toluene adsorbed. In counter part, the partial amount of n-heptane adsorbed for ESH + HEP mixtures decreases from a pressure of around 1 hPa. This decrease is all the more important as the initial mixture is enriched in mercaptan. This phenomenon accounts for a displacement of n-heptane by ethylmercaptan due to the difference between adsorption affinities of the two molecules.

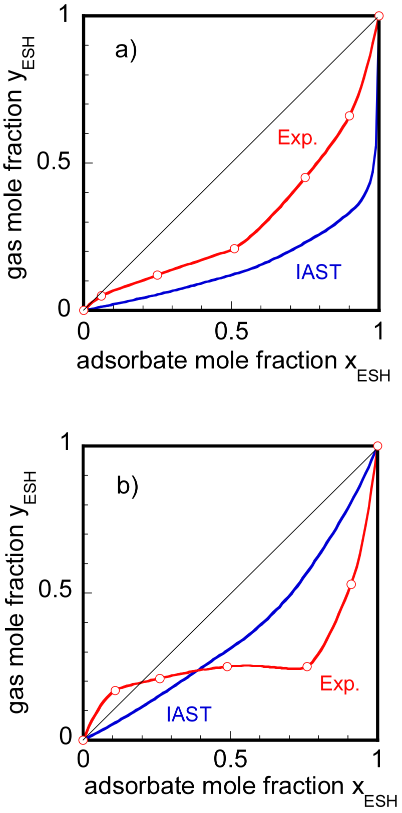

Fig. 2 y–x diagrams for adsorption of (**a**) ESH + HEP and (**b**) ESH + TOL binary mixtures on the NaX zeolite at 298 K and 0.01 hPa (*solid line*: IAST prediction; *open circles*: experiment)

On the assumption of an ideal solution, the amounts of each component adsorbed in the zeolite at constant temper-

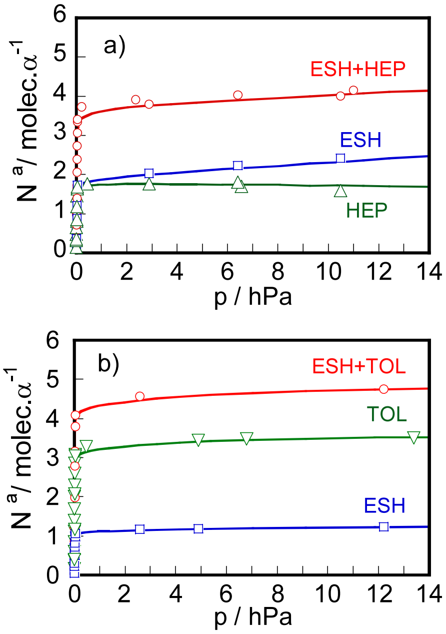

Fig. 3 Total and partial adsorption isotherms of (**a**) ESH + HEP mixture with an initial mole fraction of ESH of 0.5 and (**b**) ESH + TOL mixture with an initial mole fraction of ESH of 0.25 on the NaX zeolite at 298 K

ature and pressure must verify the so-called Lewis correlation:

$$\frac{N^{a}_{\mathrm{ESH}}}{N^{a*}_{\mathrm{ESH}}} + \frac{N^{a}_{\mathrm{HC}}}{N^{a*}_{\mathrm{HC}}} = 1$$

where N_i^a is the amount of component i adsorbed in the mixture and N_i^{a*} the amount of single component i that would be adsorbed at the same spreading pressure as that of the mixture. This relation implies that the values N_i^a and N_i^{a*} vary as a linear function of the mole fraction of ethylmercaptan y^0_{ESH} in the initial gas mixture. The dependence of the total and partial amounts adsorbed at a constant pressure of 0.2 hPa on the initial gas mixture composition is shown in Fig. 4. For both mixtures a non linear dependence is observed. The same trend is observed at other pressures. Such a result indicates that the adsorbed mixture behaves as a non-ideal solution. This deviation from ideality of the adsorbed mixture is more clearly depicted in Fig. 2, which shows the y–x diagrams. Indeed, there is a large deviation between the mole fractions predicted by the IAS Theory and those experimentally determined. Although the IAS Theory qualitatively predicts an adsorption in favour of ESH for both mixtures, it does not account for an inversion of selectivity in favour of hydrocarbon for TOL + ESH mixture enriched in toluene.

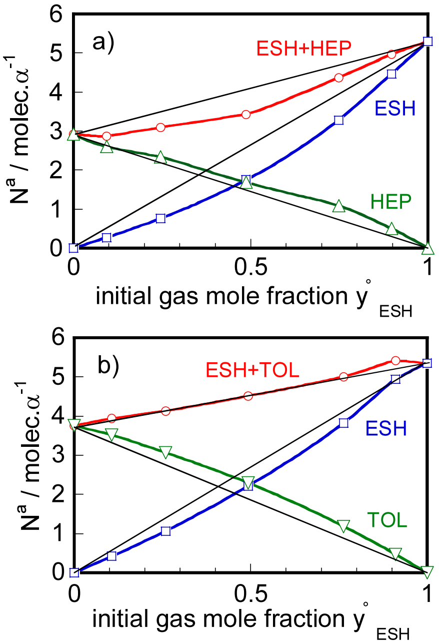

Fig. 4 Influence of the composition of the initial (**a**) ESH + HEP and (**b**) ESH + TOL gas mixtures on the total and partial amounts adsorbed on the NaX zeolite at 298 K and 0.2 hPa (*straight lines*: case of an ideal adsorbed solution)

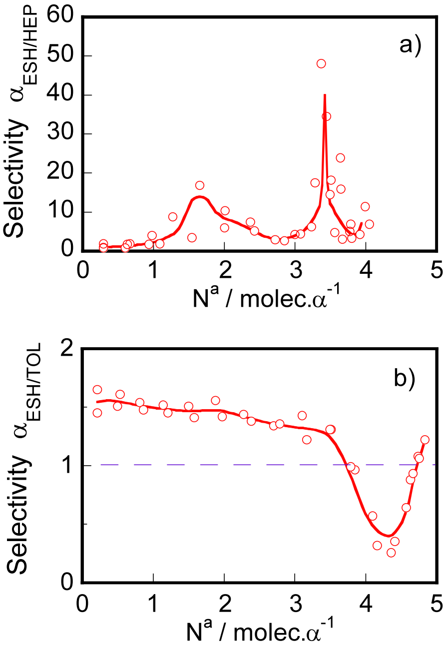

Fig. 5 Influence of the α-cage filling on the adsorption selectivity of ethylmercaptan with respect to (**a**) n-heptane or (**b**) toluene for the NaX zeolite at 298 K and for an initial mole fraction of ethylmercaptan in the gas phase of 0.25

The adsorption selectivity of ethylmercaptan with respect to n-heptane ($\alpha_{\mathrm{ESH/HEP}}$) or toluene ($\alpha_{\mathrm{ESH/TOL}}$) is the most appropriate thermodynamic value to account for the separation power of the NaX zeolite. Selectivity curves in Fig. 5 show for example the variations of $\alpha_{\mathrm{ESH/HEP}}$ and $\alpha_{\mathrm{ESH/TOL}}$ as a function of filling of α-cages for ESH + HEP and

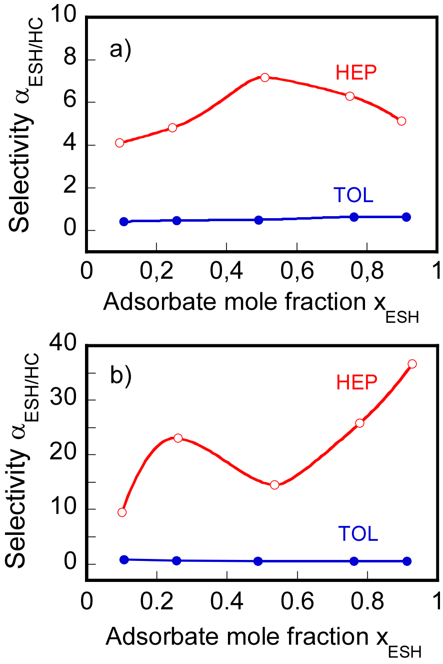

Fig. 6 Influence of the adsorbed phase composition at equilibrium on the adsorption selectivity of ethylmercaptan with respect to *n*-heptane or toluene for the NaX zeolite at a constant pressure of (**a**) 0.2 and (**b**) 4 hPa at 298 K

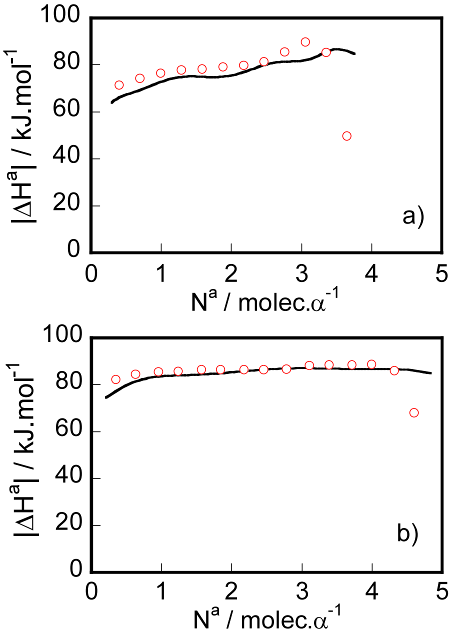

Fig. 7 Adsorption heats of (**a**) ESH + HEP and (**b**) ESH + TOL mixtures with a mole fraction of ESH in the initial gas mixture of 0.25 as a function of supercage filling of the NaX zeolite at 298 K (*open circles*: experiment; *solid lines*: values calculated from adsorption heats of single components)

ESH + TOL mixtures with an initial mole fraction of ESH in the gas phase of 0.25. Whatever the composition of the initial gas mixture, the adsorption selectivities depend on filling of the NaX zeolite. For the ESH + HEP mixture, the adsorption process is in favour of the sulphur compound at all fillings whatever the composition of initial mixture is. It is noteworthy to observe that the selectivity strongly increases at high micropore filling i.e. as the molecules are subjected to important steric hindrance in supercages (Fig. 5a). Regarding the ESH + TOL mixture, the NaX zeolite adsorbs preferentially the mercaptan over a large domain of filling up to around 4 molec·α^{-1} but $\alpha_{\mathrm{ESH/TOL}}$ is lower than $\alpha_{\mathrm{ESH/HEP}}$. Nevertheless, as filling is increased, the selectivity shows a trend to decrease, especially when the initial mixture is enriched in mercaptan. An inversion of selectivity is even observed above 4 molec·α^{-1}, indicating that toluene becomes preferentially adsorbed by the NaX zeolite. Such a removal of sulphur compound by aromatic hydrocarbon has already been observed by other authors who studied in liquid phase the competitive adsorption of toluene and thiophene on the NaY zeolite (Laborde-Boutet et al. 2006). Concerning the NaX zeolite, these authors did not find an adsorption in favour of toluene.

Figure 6 shows the dependence of adsorption selectivities $\alpha_{\mathrm{ESH/TOL}}$ and $\alpha_{\mathrm{ESH/HEP}}$ on the composition of the adsorbed phase at equilibrium and constant pressure. For the ESH + TOL mixtures the selectivity is quite constant whatever the pressure is. In return, for the ESH + HEP mixtures the selectivity greatly depends on the composition, particularly at high pressure. This is due to the larger difference in adsorption affinity between ethylmercaptan and *n*-heptane, which leads to a displacement of the alkane by the sulphur compound at high pressure.

Adsorption data of binary mixtures clearly show that the adsorbed phase behaves as a non-ideal solution. In order to determine the origin of this deviation from ideality we have measured coadsorption heats of ethylmercaptan with *n*-heptane or toluene. Therefore, some peculiar signatures characteristics of molecular interactions between the different species confined in supercages should be expected from calorimetric curves. For instance, adsorption heats of two ESH + HEP and ESH + TOL mixtures as a function of supercage filling are given in Fig. 7. Similar results are obtained with other mixtures with different compositions. Calorimetric curves exhibit the same shape as those obtained for single components. The coadsorption heats are ranged between the adsorption heats of ethylmercaptan and *n*-heptane or toluene, whatever is the composition of initial gas mixture. Heat curves do not show any particular anomalies consecutive to the appearance of specific molecular interactions in the mixture adsorbed in supercages. On the assumption of ideal adsorbed mixtures, coadsorption heats have been calculated from adsorption heats of single components by means of the following relation:

$$\Delta H^{a}_{\mathrm{ESH+HC}} = x_{\mathrm{ESH}} \Delta H^{a*}_{\mathrm{ESH}} + x_{\mathrm{HC}} \Delta H^{a*}_{\mathrm{HC}},$$

where $\Delta H_i^{a^*}$ denotes the adsorption heat of single component i at the same filling as that of the mixture. These experimental data are reported in previous papers (Weber et al. 2005; Benoit et al. 2005). A rather good correlation is obtained between experimental and calculated coadsorption heats. Therefore, the deviation from ideality of adsorbed mixtures may be ascribed to entropic rather than enthalpic effects.

Such a conclusion was drawn from previous studies devoted to the separation of xylene isomers by selective adsorption on faujasite zeolites exchanged with barium (Cottier et al. 1997). Indeed, the coadsorption process always occurred in favour of *p*-xylene at high supercage filling although (i) adsorption isotherms of single *p*-xylene and *m*-xylene were perfectly superimposed; and (ii) the corresponding adsorption heats were quite the same. Moreover, the presence of water molecules preadsorbed in supercages largely influenced the selectivity by generating steric hindrance that disturbed the packing of molecules into cavities (Moïse and Bellat 2005). We already realized at this time that the entropic effect could be as important as the energetic effect. This is especially the case for molecules like aromatic hydrocarbons, which are confined in restricted environments like supercages of faujasite zeolites. Other examples of selective adsorption process governed by entropic effect, such as the adsorption of iso-alkanes in SAPO-5, MCM-22 or ZSM-5, have been recently reported in the literature (Denayer et al. 2006). Many similarities are observed between these systems and the one of the present study. To conclude, the competitive adsorption of a mercaptan with hydrocarbons on the NaX zeolite must be considered as a process where the selectivity is controlled by entropic packing effects, particularly at high degree of pore filling.

4 Conclusion

This basic study shows that the NaX zeolite could be a good adsorbent for a deep desulphurization of natural gas by selective adsorption. Indeed, this nanoporous material exhibits a strong adsorption affinity for ethylmercaptan. When the sulphur compound is in the presence of *n*-heptane its adsorption selectivity is not affected. The selectivity remains in favour of mercaptan whatever are the filling and the composition of the mixture. Nevertheless, another behaviour is observed when ethylmercaptan is coadsorbed with toluene. Indeed, if the adsorption process is also in favour of mercaptan at low and moderate loadings ($N^a < 4$ molec·α^{-1}), conversely an inversion of selectivity occurs at high loading. Then, the NaX zeolite adsorbs preferentially the aromatic hydrocarbon. For industrial application this could be a disadvantage. This implies that the NaX zeolite should not work at too high filling and should be thereafter frequently regenerated. Moreover, even after amine washing and glycol dehydration, the natural gas still contains traces of water and other impurities, which can influence the selectivity. This study must be extended to more complex mixtures in order to know if this adsorbent can really be used to perform a deep desulphurization at the industrial scale.

On the other hand, the analysis of coadsorption data indicates that the adsorbed mixture does not behave as an ideal solution: the adsorption selectivity depends on filling and composition and the IAS Theory is not able to reproduce the experimental values of selectivity. Moreover, the selective adsorption process is essentially governed by entropic effects due to steric hindrance of molecules in confined spaces. That being the case, the nature of extra-framework cations must be the key factor of selectivity. The desulphurization of natural gas by adsorption on exchanged X zeolites is in progress.

5 Nomenclature

N^a: amount of mixture adsorbed, molec·α^{-1}
N_i^a: amount of component i adsorbed in the mixture, molec·α^{-1}
$N_i^{a^*}$: amount of single component i adsorbed at the same spreading pressure as for the mixture, molec·α^{-1}
p: pressure of gas at equilibrium, hPa
p_s: saturated vapour pressure at the adsorption temperature, hPa
p/p_s: relative pressure
T: temperature of adsorption, K
V^a: volume of liquid adsorbate, cm^3·g^{-1}
$V^a_{\max i}$: maximal volume of liquid adsorbate determined by the Dubinin-Radushkevich model, cm^3·g^{-1}
x_i: mole fraction of component i in the adsorbed phase
y_i^0: mole fraction of component i in the initial gas mixture
y_i: mole fraction of component i in the gas phase at equilibrium
θ: loading rate of adsorption
$\Delta H^a_{\mathrm{ESH}+HC}$: differential molar enthalpy of adsorption of mixture, kJ·mol^{-1}
$\Delta H_i^{a^*}$: differential molar enthalpy of adsorption of single component i at the same filling as for the mixture, kJ·mol^{-1}
$\Delta H_\infty^{a^*}$: differential molar enthalpy of adsorption of single component i extrapolated at zero filling, kJ·mol^{-1}
$\Delta S_\infty^{a^*}$: isosteric entropy of adsorption of single component i extrapolated at zero filling, J·K^{-1}·mol^{-1}
$\Delta G_\infty^{a^*}$: differential molar Gibbs energy of adsorption of single component i extrapolated at zero filling, kJ·mol^{-1}
$\alpha_{\mathrm{ESH/HC}} = x_{\mathrm{ESH}} \cdot y_{\mathrm{HC}}/x_{\mathrm{HC}} \cdot y_{\mathrm{ESH}}$: adsorption selectivity of ethylmercaptan with respect to the hydrocarbon HC

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