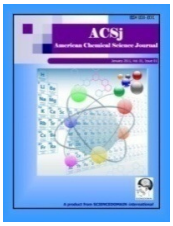

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Single and Competitive Adsorption of Linear and Branched Paraffins over Silicalite: Thermodynamic and Kinetic Study

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Authors' contributions

This work was carried out in collaboration between all authors. Author AHY designed the study, performed the statistical analysis, wrote the protocol, and wrote the first draft of the manuscript. Author IGB managed the analyses of the study. Authors IGB and SM managed the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

A thermodynamic and kinetic study was realized by competitive and non-competitive adsorption for the separation of n-hexane isomers. Two mixtures of n-C6/3MP and 3MP/23DMB were studied. For the single component adsorption the maximal adsorption capacity were close for the different isomers while the diffusivity for the 23DMB isomer was outstandingly low due to steric hindrance. For the competitive adsorption we had from an initial proportion of 50% for each sorbate an adsorption of 34% and 66% respectively for 3MP and n-C6. For the 3MP/23DMB mixture we had an adsorption of 62% and 38% for 3MP and 23DMB respectively. For the two mixtures the selectivity of the zeolite increased with the adsorption capacity and decreased after the values of 0.63 mmol/g and 1.65 mmol/g respectively for the 3MP/23DMB and n-C6/3MP mixtures. The adsorption rates were lower in the case of competitive adsorption and a kinetic separation could be envisaged for the improvement of the octane number.

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1. INTRODUCTION

For the last decade the legislation for the protection of environment became more drastic all over the world. The restrictions concerns particularly the refinery industry which has to produce cleaner and more efficient gasoline. With the prohibition of some additives in the composition of gasoline the branched alkanes appeared as very good solution for the production of clean and efficient gasoline. In many refineries isomerization units convertes normal paraffins to higher-octane number isoparaffins but the convertion rate never riches 100% and the remaining normal and monobranched paraffins reduce the octane number of the final product. So the separation of dibranched paraffins from mixtures of linear and branched paraffins is of a great interest for the refinery industry. Such separation is difficult by classic methods as distillation, crystallization or solvent extraction because the physical properties of the considered hydrocarbons are very close. Due to there molecular shape selectivity zeolites are well indicated for this separation. 5A zeolite have been widely studied as selective adsorbent for the separation of alkane isomers [1] but silicalite, MFI, FER, MOR, SAPO-5 or BEA type zeolites have received and increasing interest these last years [2-28]. The present work deals with the thermodynamic and kinetic characteristics of the adsorption of hexane isomers on silicalite. The experiments were realized by thermogravimetry or volumetry for single and mixed paraffins.

2. EXPERIMENTAL DETAILS

2.1 Adsorbent and Reagents

The silicalite was provided by Zeolyst International. The Si/Al ratio determined by elemental analysis was about 504 [29]. The BET surface and the porous volume were determined by nitrogen adsorption-desorption experiments performed at 77 K using a TRISTAR Micromeritics instrument. The BET surface was about 368 m^2/g and the total porous volume was about 0.2 cm^3/g. The crystals were essentially of a spherical shape of 1-3 µm. The crystallinity, determined by XRD, was about 98%.

n-hexane (n-C_6), 3-methylpentane (3MP) and 2,3-dimethylbutane (23DMB) with a purity of 99,9% were provided by Fluka Chemie AG. The physical characteristics of the sorbates are listed in Table 1.

Table 1. Physical characteristics of the paraffins

Sorbates	Critical diameter (nm)	Length (nm)	Vapor pressure 298 K (bar)	Octane number RON
n-C6	0.49	1.0	0.201	31
3MP	0.54	0.94	0.253	76
23DMB	0.58	0.81	0.425	96

2.2 Procedures

The single component adsorption performed by thermogravimetry was realized at 298 K using a SETARAM microbalance. The zeolite samples (ca. 65 mg) were out-gassed under secondary vacuum at 623 K for 12 h prior to sorption measurements and cooled down to 298 K. The pressure of the sorbate was then increased step by step to determine the adsorption isotherm. The adsorption isotherms were modelized by the Langmuir equation (1).

$$\frac{P}{M} = \frac{1}{K.M_\infty} + \frac{P}{M_\infty} \quad (1)$$

Where

P = partial pressure of the sorbate
M = amount of paraffin adsorbed per gram of zeolite at the pressure P
M_∞=maximum adsorption capacity per gram of zeolite
K = Langmuir adsorption coefficient

The diffusion in the crystals was assumed to follow Fick's equations. The mathematical solution for the transient diffusion equation involving a spherical particle in terms of uptake of sorbate by the solid assumes the well-known form given by Crank [30]:

$$\frac{M_t}{M_\infty} = 1 - \frac{6}{n^2}\sum_{n=1}^{\infty}\left[\frac{1}{n^2}.\exp(-\frac{n^2\pi^2 D}{r^2})\right] \quad (2)$$

Where M_t and M_∞ are respectively the adsorbed amounts at the time t and at the sorption

equilibrium, D is the diffusivity and r is the radius of the crystallites.

A simplified and convenient solution for short times is [31]:

$$\frac{M_t}{M_\infty} = \frac{6}{\sqrt{\pi}}\sqrt{\frac{D}{r^2}t} \quad (3)$$

For long times, the solution of the equation is [31]:

$$\frac{M_t}{M_\infty} = 1 - \frac{6}{\pi^2}.\exp\left(-\pi^2\frac{Dt}{r^2}\right) \quad (4)$$

The diffusion can be affected by heat transfer or by a wide distribution of crystal size in the sample. The short time response is less affected by these parameters, so it was used in the present paper for the assessment of diffusivities.

The volumetric experiments were performed for both single and competitive adsorption. The diagram of experimental set-up is presented on Fig. 1.

The volumetric set-up was constituted by two flaskes containing the solutions of linear or branched paraffins.

The same pressure of two sorbates was expanded to a mixing chamber for competitive adsorption experiments. The alkane mixture was homogenized during four hours. The adsorption experiments were performed at 298 K. The zeolite sample (ca. 150 mg) was out-gassed under secondary vacuum at 623 K for 12 h prior to the sorption measurements and cooled down to 298 K. A first increment of pressure of the homogenized mixture was slackened to the volume V_1. A small part of this gas was analysed by GC to determine the composition of the mixture before adsorption. The residual gas pressure in the V_1 volume was recorded. The mixture of sorbate was then slackened in the V_r volume. A progressive decrease of the pressure occurred due to the adsorption on the zeolite. The weight of the zeolite was recorded versus the time for the determination of diffusivities. When the pressure was stabilized in the V_t volume (with $V_t = V_1 + V_r$) the V_r volume was isolated and the residual pressure in the V_1volume was analyzed by GC to determine the composition of the mixture after adsorption. The new final pressure in the V_1 volume was recorded and a small increment of the mixture was expanded in the V_1 volume.

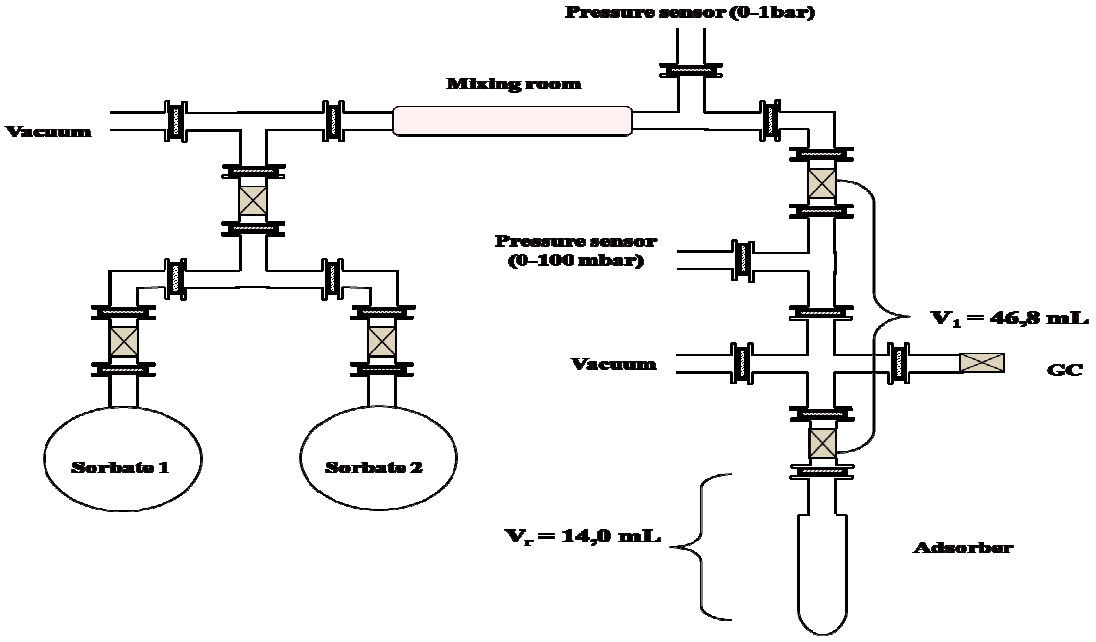

Fig. 1. Experimental set-up for volumetric measurements

The gas composition in the V_1 volume was analyzed and the adsorption experiments continued until the complete isotherm was obtained.

The single component adsorption was performed in the same conditions than competitive adsorption and the gas mixtures were replaced by single sorbates.

For competitive adsorption experiments the gas compositions were determined by Varian Star 3400 GC equipped with a DB1 column and a FID detector.

For single component adsorption, by assuming that gases were perfect the following relations were applied:

For the first equilibrium (k = 1):

$$n_{ads}(mol) = n_i(gas) - n_f(gas) = \frac{P_1 V_1}{RT} - \frac{P_t V_t}{RT} \quad (5)$$

For the $(k+1)^{th}$ equilibrium, the amount of adsorbed molecules became :

$$n_{ads}(mol) = n_f^{th}(gas)\frac{V_r}{V_t} + \frac{P_1' V_1}{RT} - \frac{P_t' V_t}{RT} \quad (6)$$

Where

- n_{ads} = amount of adsorbed molecules by the zeolite (mmol/g)
- n_f (gas)= final amount of molecules in gas phase in the V_t volume (mmol/g)
- n_i (gas) = initial amount of molecules in gas phase in the V_1 volume (mmol/g)
- P_1 = gas partial pressure in the initial V_1 volume
- P_t = gas partial pressure in the total V_t volume
- V_r = volume of the adsorber
- V_t = V_1 + Vr
- P'_1 = gas partial pressure in the V_1 volume for the $(k+1)^{th}$ point
- P't = gas partial pressure in the V_t volume for the $(k+1)^{th}$ point

For the adsorption of mixtures the adsorbed amounts for each constituent *i* were given by the following relation (7) for the k^{th} equilibrium.

$$n_i^a(k+1) = \left[\frac{V_1}{RT}(y_{1i}(k+1)P_1 - y_i(k+1)P_t) + \frac{V_r}{RT}(y_i(k)P_1 - y_i(k+1)P_t)\right] + n_i^a(k) \quad (7)$$

Where $y_i(k)$ and $n_i^a(k)$ were respectively the molar fraction of the *i* constituent at equilibrium in the gas phase, and the amount of the *i* constituent for th k^{th} adsorption. The adsorbed amounts were also expressed in molecules per unit cell using the relation (8):

$$N_i^a(k) = n_i^a(k).M.10^{-3} \quad (8)$$

Where M was the molar mass of the zeolite.

The selectivity of the *i* constituent in comparison with the *j* constituent was given by the relation (9).

$$\alpha_{i/j} = \frac{x_i(k)y_j(k)}{x_j(k)y_i(k)} \quad (9)$$

In this relation x_i represented the molar fraction of the *i* constituent adsorbed at the k^{th} equilibrium.

$$x_i(k) = \frac{N_i^a(k)}{\sum_i N_i^a(k)} \quad (10)$$

The diffusion followed the Fick's law and by simplification the relation (11) was used for short times approximations:

$$\frac{M_t}{M_\infty} = 6\frac{1+K}{K\sqrt{\pi}}\sqrt{\frac{Dt}{r_0^2}} \quad (11)$$

Where K was a ratio determined by the amount of sorbate in the gas phase and the amount of sorbate at equilibrium.

For long times approximations the relation (12) was used.

$$\ln\left(\frac{M_\infty - M_t}{M_\infty - M_0}\right) = \ln\left[\frac{6K(K+1)}{9(K+1)+P_1^2K^2}\right] - \frac{P_1^2 Dt}{r_0^2} \quad (12)$$

3. RESULTS AND DISCUSSION

3.1 Single Component Adsorption Performed by Gravimetry and Volumetry

In the case of non-competitive adsorption the isotherms were fitted by the Langmuir model. Furthermore the adsorption experiments performed by gravimetry and volumetry exhibited that the thermodynamic characteristics determined by these two methods were much close Tables 2a and 2b.

Table 2a. Thermodynamic characteristics for the non competitive adsorption of paraffins determined by volumetry

Sorbates	M_∞ (mmol.g^{-1})	M_∞ (molecules.UC^{-1})	K (mbar^{-1})
n-C_6	1.33	7.61	0.2210
3MP	1.20	6.90	0.0520
23DMB	1.31	7.55	0.0509

Table 2b. Thermodynamic characteristics for the non competitive adsorption of paraffins determined by gravimetry [32-33]

Sorbates	M_∞ (mmol.g^{-1})	M_∞ (molecules.UC^{-1})	K (mbar^{-1})
n-C_6	1.32	7.6	0.2140
3MP	1.19	6.8	0.0524
23DMB	1.31	7.5	0.0509

Due to steric hindrance the maximal adsorption capacity was slightly higher for n-hexane compared to branched isomers 3MP and 23DMB. Basing upon the Langmuir constants it appeared that linear isomer was better adsorbed than branched isomers. Furthermore the values of Langmuir K constants were much close for the branched isomers (3MP and 23DMB). So the thermodynamic separation of n-hexane from the branched isomers could be more easily envisaged than the thermodynamic separation of monobranched (3MP) and dibranched (23DMB) isomers.

Kinetic studies were performed for initial partial pressure of the sorbate equal to 100 mbar. It appeared from Fig. 2 that the kinetics was very different for the dibranched isomer (23DMB) compared to the other isomers (n-C_6 and 3MP).

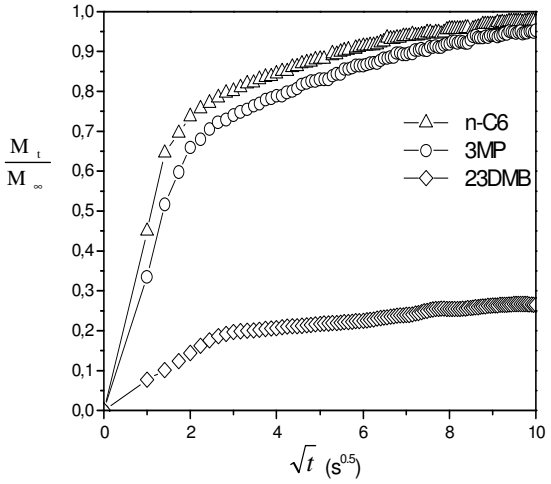

Fig. 2. Short times diffusivities for single component adsorption of hexane isomers on silicalite at 298 K

The short times diffusivities calculated from equation (11) are reported in Table 3.

Table 3. Short times diffusivities for hexane isomers in non competitive volumetric adsorption

Sorbates	n-C_6	3MP	23DMB
Short time diffusivities (s^{-1})	6.95 10^{-3}	4.39 10^{-3}	2.43 10^{-4}

The adsorption kinetics for n-C_6 was respectively 30 times and 1.6 times faster than the adsorption kinetics for 23DMB and 3MP. The steric hindrance appeared to be the key factor influencing the diffusivity of the sorbates. Thus it was highlighted that the dibranched isomer (23DMB) could be easily separated from the other isomers by a kinetic techniques.

3.2 Competitive Adsorption Performed by Volumetry

The competitive adsorption was studied for two types of mixtures: n-C_6/3MP mixture and 3MP/23DMB mixture. The mixtures contained 50% of each sorbate. The adsorption isotherms are represented on Figs. 3a and 3b.

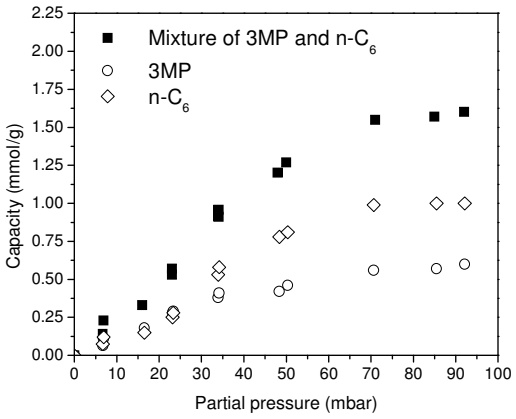

Fig. 3a. Adsorption isotherms for the mixture of n-C_6/3MP on silicalite at 298 K

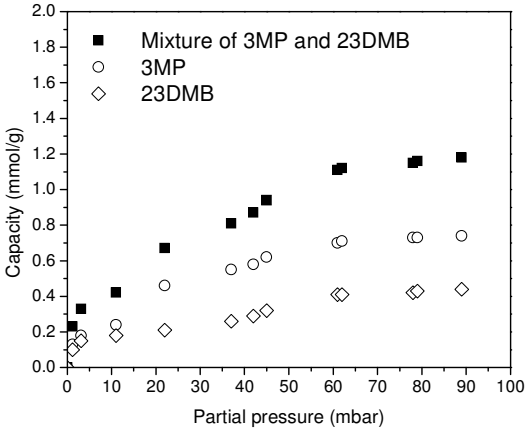

Fig. 3b. Adsorption isotherms for the mixture of 3MP/23DMB on silicalite at 298 K

The adsorption isotherms were fitted by the model of Langmuir. The maximal adsorption capacities and the Langmuir constants are reported in Table 4.

Table 4. Langmuir parameters for competitive adsorption of paraffins determined by volumetry

Sorbates	n-C_6/3MP mixture		3MP/23DMB mixture	
	M_∞ (mmol.g^{-1})	K (mbar^{-1})	M_∞ (mmol.g^{-1})	K (mbar^{-1})
n-C_6	0.60	0.0547	-	-
3MP	1.03	0.0319	0.91	0.0525
23DMB	-	-	0.73	0.0174

It appeared from Table 4 that the maximal adsorption capacities were lower in the case of competitive adsorption when comparing to single adsorption data Table 2a. Indeed in the case of competitive adsorption [29] the sorbate uptake may be lower due to steric hindrance between adsorbed molecules.

The Fig. 3a exhibited that for the low partial pressures (inferior to 30 mbar) the adsorption capacities for n-C_6 and 3MP were very close and the zeolite was not selective for any sorbate. For the values of pressures between 30 mbar and 60 mbar a preferential adsorption of n-hexane was encountered. Beyond the partial pressure of 60 mbar the maximal adsorption capacity was achieved for the two sorbates. From initial sorbates proportion of 50% in the gas mixture 34% and 66% of 3MP and n-C_6 were respectively adsorbed in the silicalite.

The Fig. 3b showed a preferential adsorption of 3MP from the lower partial pressures. The maximal adsorption capacity was achieved when the partial pressure was equal to 80 mbar and the adsorbed mixture consisted in 62% and 38% of 3MP and 23DMB respectively.

From the Fig. 4a presenting the selectivity of n-C_6 in comparison with 3MP versus the adsorption capacity of the n-C_6/3MP mixture it appeared that the zeolite was selective when the adsorption capacities were in the range of 0.7 to 2.2 mmol/g.

When considering the mixture of 3MP and 23DMB isomers the Fig. 4b exhibited that the zeolite was selective for adsorption capacities in the range of 0.45 mmol/g to 0.7 mmol/g.

The kinetic study of the mixtures was performed by mixing 50 mbar of each sorbate. So the initial pressure in the system was 100 mbar and the results could be compared to those obtained in the case of single component adsorption. The sorbates uptake versus the time is represented on Figs. 5a and 5b.

The kinetics were in the range of several minutes when for the single component adsorption it was about a few seconds. One explanation for that is the competition between isomers for the access to the porosity of the zeolite.

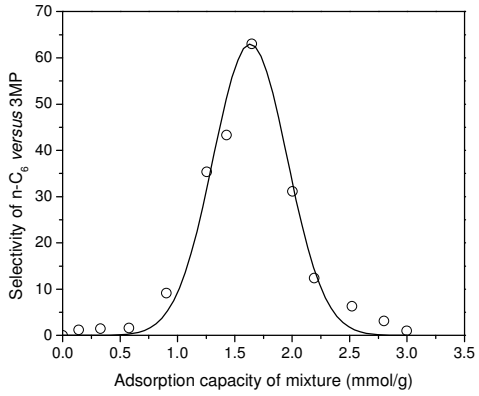

Fig. 4a. Selectivity of n-C_6 in comparison with 3MP versus the adsorption capacity of the n-C_6/3MP mixture for silicalite at 298 K

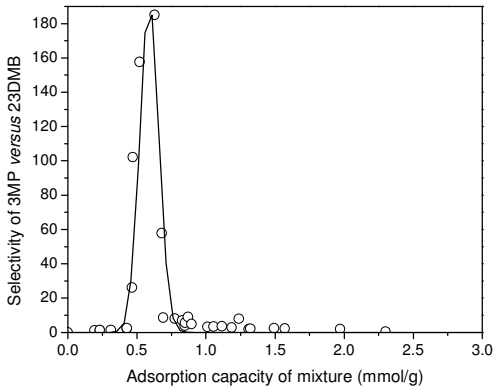

Fig. 4b. Selectivity of 3MP in comparison with 23DMB versus the adsorption capacity of the 3MP/23DMB mixture for silicalite at 298 K

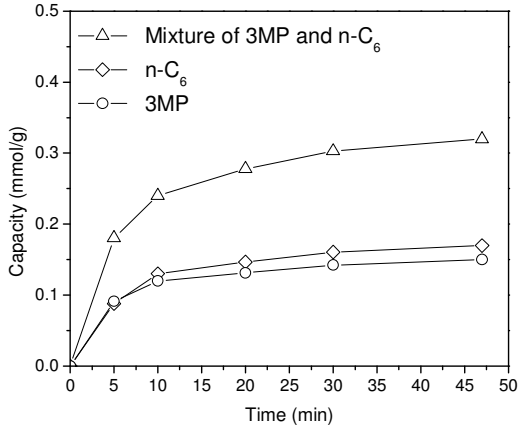

Fig. 5a. Co-diffusion of the n-C_6/3MP mixture on silicalite at 298 K

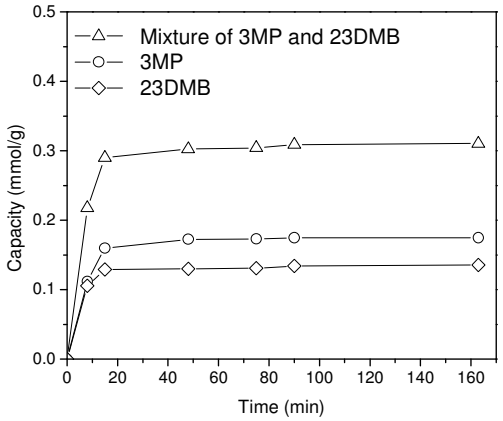

Fig. 5b. Co-diffusion of the 3MP/23DMB mixture on silicalite at 298 K

The Fig. 5a showed that before 10 min there was no preferential adsorption for n-C_6 nor 3MP but after 10 minutes a preferential adsorption for n-C_6 was noticed. The same trend was observed for 3MP/23DMB mixture Fig. 5b. Indeed before 15 minutes similar adsorption capacities were noted for the two isomers and after 15 minutes a preferential adsorption of 3MP occurred. Thus these experiments exhibited that a kinetic separation of mixtures of normal and branched paraffins by adsorption over silicalite could be envisaged. Furthermore the possibility of a kinetic separation between monobranched isomer (3MP) and dibranched isomer (23DMB) was feasible.

4. CONCLUSION

Both single and competitive adsorption of n-hexane isomers were performed over silicalite. The adsorption data recovered by gravimetric and volumetric methods were very close. From single component adsorption experiments it was observed that the 23DMB isomer could be isolated by both thermodynamic and kinetic separations. The competitive adsorption experiments showed that silicalite was selective for the adsorption capacities in the range of 0.7 mmol/g to 2.2 mmol/g for the n-C_6/3MP mixture and the range of 0.45 mmol/g to 0.7 mmol/g for the 3MP/23DMB mixture. Anyway the adsorption

capacity and kinetics were lower for the branched isomers. By comparison with the single component adsorption the kinetics was very low for the competitive adsorption because of the competition between the isomers for the access to the porosity of the zeolite. Consequently a kinetic separation could be envisaged for the improvement of the octane number of gasoline coming from isomerization units. For an initial amount of 50% for each sorbate in the gas mixture, 34% and 66% of 3MP and n-C_6 were respectively adsorbed in the case of competitive adsorption. For the 3MP/23DMB mixture a preferential adsorption of 3MP was encounterred; the proportions of the adsorbed isomers were respectively 62% and 38% for 3MP and 23DMB.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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