Determination of Adsorption Characteristics of Volatile Organic Compounds Using Gas Phase FTIR Spectroscopy Flow Analysis

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1. Introduction

Adsorption is an important operation for gas mixture separation and purification (Yang et al.1987). Thus, measurement of adsorption capacity is crucial for correct designing and operating of adsorption facilities. The heat of adsorption is also an important adsorption characteristic usually needed if adsorbent regeneration and/or adsorbate recovery is targeted (Do, 1998). Accurate knowledge of this thermodynamic parameter is of interest because the heat effects involved during adsorption and/or desorption may induce significant local warming to start combustion of either the adsorbate or the adsorbent itself, particularly, when activated carbon is used (EPA, 1998).

In case of physical adsorption, the involved heat effects might be determined with the isosteric method based on Clausius–Clapeyron equation using adsorption isotherms data at various temperatures (Sircar et al.1999). However, it is to be noted that the isosteric method is limited only to physisorption processes involving relatively low heat of adsorption and for equilibrium reached within accessible range of pressure and temperature (Ranke et al.2002). Under equilibrium, the physically adsorbed species are trapped in different potential wells predisposed within the accessible porosity. During desorption, the species adsorbed in more easily accessible porosity desorbs first. As for those adsorbed in the less accessible porosity, one has to supply required thermal energy for their transfer into gas phase. This energy could be estimated by TPD method using a relatively inexpensive and simple experiment to set up and to run (Cvetanovic et al.1967).

This chapter is devoted to the description of an easy and efficient method based on the application of gas phase Flow FTIR spectroscopy analysis for determination of adsorption characteristics of volatile organic compounds. As adsorbent beds are usually operated under dynamic conditions, the adopted analytical approach is based on gas phase composition monitoring at reactor outlet during adsorption/ desorption experiments carried out under dynamic regime. This method permits further simultaneous detection of new IR bands that may originate from adsorbate dissociation during adsorption or desorption.

Although, Infra Red (IR) spectroscopy is generally used for compounds identification based on measurement of IR radiation resulting from molecular vibration perturbation. The application of Beer's law allows quantitative exploitation of IR spectra using preliminary calibration with known composition. Therefore, the technique was found to be adequate for transient signals monitoring and quantification owing to faster response to instantaneous change in IR bands positions and intensities due to rapid spectra acquisitions allowed by FTIR instrumentation (Chafik et al, 1998). In addition, the high transparency of gases and the low background values achieved, allows detection of lower species concentration in the gas stream. Further increase of analytical sensitivity might be obtained with multiple-pass gas cell.

In the present study, we show an example of the application of this experimental methodology to the investigation of adsorption characteristic of local bentonite clay. Adsorption / desorption experiment were performed at laboratory scale with model-contaminated gaseous stream containing o-xylene that has been selected as representative VOC because it is environmentally relevant regarding industrial concern. The work aimed to help further development of low cost materials involved in environmental engineering control.

2. Experimental

2.1 Adsorbent

The clay tested in the present work comes from deposits located in the north of Morocco (Nador area). The results corresponding to its textural and mineralogical composition have been published (Harti et al, 2007). The clay was found to be a mixture of phases, namely, opal, montmorillonite, kaolinite, muscovite, topaz, rutile, calcite, dolomite, suggesting a bentonite type clay. Textural studies perfomed with N_2 adsorption desorption at 77 K have shown a BET specific surface area of 79 $m^2 g^{-1}$, and a negligible micropore volume of 0.002 $cm^3 g^{-1}$ as compared with total pore volume of 0.205 $cm^3 g^{-1}$. The pore size distribution obtained following BJH method revealed the presence of a wide pore size distribution in the mesopore range with a significant contribution of pores widths between 8 and 50 nm.

2.2 Adsorption and desorption experiments

Adsorption/desorption experiments were performed under dynamic conditions at atmospheric pressure using the experimental apparatus as reported elsewhere (Zaitan et al.2005). The bentonite adsorptive properties were investigated with respect to O-xylene vapour. Prior to adsorption, a model mixture with a given concentration of o-xylene vapour in nitrogen flow, was prepared by means of a saturator connected to a condenser that was immersed in a thermostatically controlled bath. This temperature was carefully checked so as to maintain constant *o*-xylene vapour pressure and consequently keep the *o*-xylene concentration unchanged. The resulting concentration is expressed as molar fraction (or partial pressure; P/P_0), where P is the vapour pressure of *o*-xylene obtained from Antoine equation and P_0 the atmospheric pressure taken as 760 Torr. Hence, fixing condenser temperature between -8 and 40°C permitted obtaining a value of concentration at reactor inlet (C_{in}) in a range of 700 - 11000 ppm (Zaitan et al, 2006).

2.3 Determination of adsorbed and desorbed amounts

The o-xylene concentrations at reactor outlet during adsorption and desorption were monitored with a FTIR spectrometer (Jasco 410, resolution 4 cm^{-1}), using a Pyrex gas cell equipped with CaF$_2$ windows. The quantitative analysis is facilitated by FTIR instrumentation and programs that allows high frequency spectra acquisition and manipulation (substraction, multiplication, smoothing). This operation is particularly easy when there are no IR bands overlapping and the application of Beer–Lambert law permits relating IR bands area to concentration. In the present work, the quantitative treatment was achieved by integrating o-xylene IR bands located between 2600 and 3200 cm^{-1}. Preliminary calibration with o-xylene/N$_2$ mixtures of known composition was carried out using reactor by pass, in order to correlate bands area with concentration. Figure 1 shows the calibration curve representing the integral of o-xylene IR bands area between 2600 and 3200 cm^{-1} as function of concentration. The FTIR response was found to produce linear plot in the studied concentration range and its accuracy was cheeked over 3 experiments and represented as mean value.
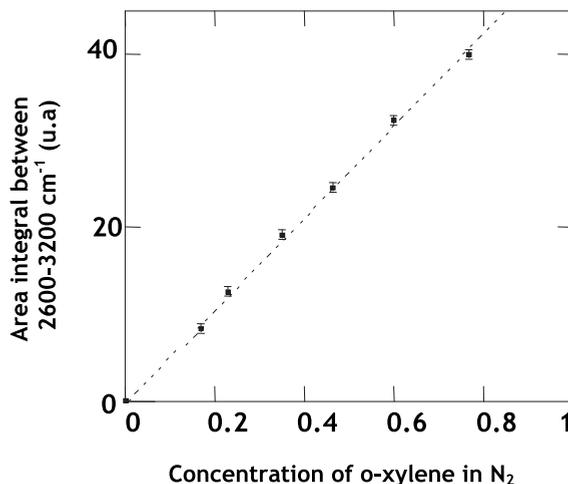

Fig. 1. Calibration curve o-xylene IR band area integral (2600-3200 cm^{-1}) versus its concentration in N$_2$ (molar fraction %)

The sample pre-treatment as well as adsorption and desorption experiments were performed with a flow rate of 100 cm^3 min^{-1} that was passed through a quartz reactor (U-type) containing 1 g of bentonite meshes. The sample was first pre-treated under N$_2$ flow at 473 K for 30 minutes than adsorption was carried out using the model mixture flow until saturation was reached in order to obtain breakthrough curves. The gas mixture was switched again to pure N$_2$ flow, to proceed with isothermal desorption until o-xylene concentration at the reactor outlet reached zero. This step was followed by a subsequent linear heating in order to perform Temperature Programmed Desorption (TPD) experiment.

The monitoring of o-xylene IR bands during adsorption at 300 K shows gradual increase of IR bands until equilibrium was reached, which corresponds to adsorbent saturation (Fig2, part A). Following this step, the gas mixture was switched to pure nitrogen flow (0.36 % o-xylene/N$_2$→N$_2$), to perform isothermal desorption giving rise to gradual decrease of IR
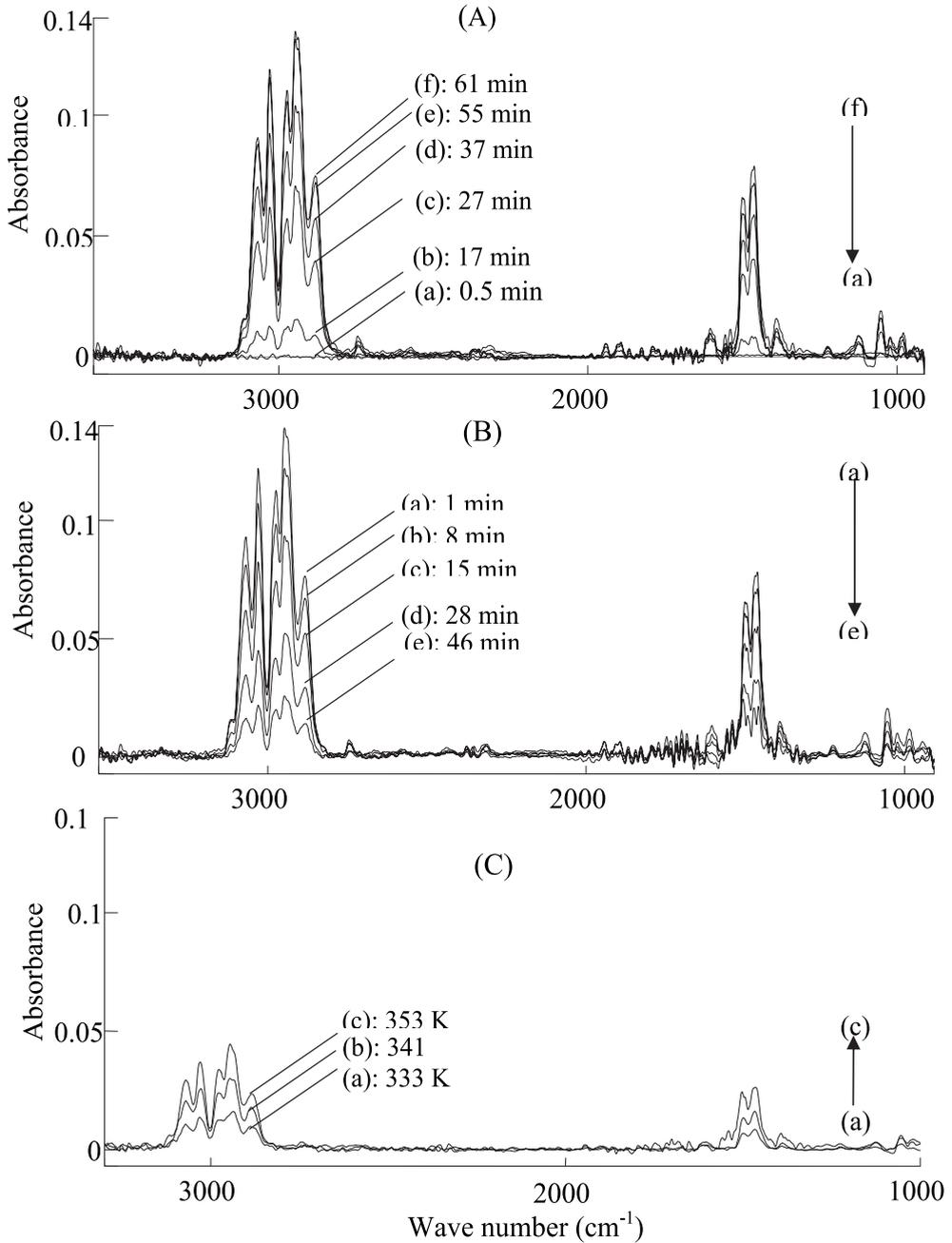
Fig. 2. Evolution of o-xylene IR bands during the following successive experiments: A; Isothermal adsorption at 300K, B; isothermal desoprtion and TPD under N$_2$ at linear heating rate of 5K/min.

bands with time, as shown in part B. The sample was, than, linearly heated up to 473K to carry out TPD experiment under N_2 flow (part C of Figure 2). It is to be noted, that the recorded FTIR spectra do not reveal any new species formation originating from o-xylene transformation due to catalytic activity. This information provided by the use of FTIR analysis technique can be considered as an advantage of the experimental approach.

The evolution of *o*-xylene concentration at the reactor outlet (C_{out}) obtained from FTIR spectra, permitted the monitoring of adsorbent loading as function of time during adsorption (i.e. breakthrough curve) and desorption processes.

Figure 3 shows the profile of the variation of *o*-xylene concentration in the gas flow at reactor outlet, represented as relative values (C_{out}/C_{in}), during the aforementioned cycle of successive steps (adsorption at 300K until saturation followed by isothermal desorption than Desorption with Programmed Temperature).
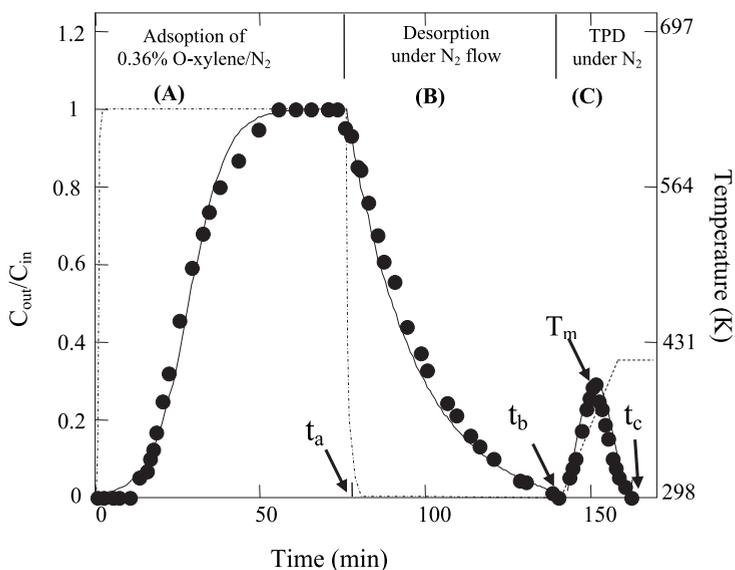
— ·— without adsorbent
------- heating profile during TPD experiment

Fig. 3. Profile of the variation of *o*-xylene concentration in the gas flow at reactor outlet, represented as relative values (C_{out}/C_{in}), during a cycle of successive steps; adsorption performed with a mixture of 0.36% xylene in N_2 at 300K until saturation followed by isothermal desorption than Temperature Programmed Desorption carried out with a linear heating rate of 5K/min

It is to be noted that the adsorption/ desorption experiments carried out in the present work with samples of 1g of bentonite compressed as small meshes contained in a quartz microreactor (type U with an internal volume ≈ 1 cm^3). The experiments carried out under atmospheric pressure (760 torr) using total flow rate of 100 cm^3/min resulted in a residence time <1 sec. Under these conditions the flow pass through the sample in mode 'plug flow' and since small amounts are involved in adsrorption or desorption experiments, the reactor

could be considered operating under differential mode as Continuous flow Stirred Tank Reactor (CSTR). This approach permit creation of appropriate and accurate conditions that avoid limitation due to mass and heat transfer within the adsorbent particles in the reactor bed (Zaitan et al, 2008). Consequently, the experiments could be considered as occurring under isothermal conditions in which the pressure drop as well as the variation in fluid velocity between the reactor inlet and outlet were considered negligible. Thus, the adsorbed amount can be calculated using the mass balance equation (Ruthven, 1984).

$$n_{ads} = \frac{FC_{in}}{m}\left[t_a - \int_0^{t_a} \frac{C_{out}}{C_{in}} dt \right] \tag{1}$$

Where n_{ads} is the adsorbed mole number of toluene at saturation (adsorption capacity in mol/g , C_{in} and C_{out} the molar fractions of toluene at reactor inlet and outlet, m is the absorbent mass, t_a the saturation time and F the gaseous molar flow rate.

The adsorbed amount measured from breakthrough curve is generally used to indicate adsorbent performance in terms of a given constituent removal from a flowing stream. In the present study, the adsorption capacity is obtained by integration of the breakthrough curve, according to the equation (1) and considering the curve corresponding to the reactor response in the absence of solid. Accordingly, the numerical integration of the breakthrough curve using MathCAD software yields to a total adsorbed amount of 420 μmol g^{-1}, for adsorption carried out with a flow containing 0.36% o-xylene in N_2 (Fig. 3 part A). This amount is lower than the adsorption capacity of 1958 μmol g^{-1} reported in our previous work for SiO_2 Degussa (200 m²g^{-1}) (Zaitan et al 2005). In comparison with the literature, substantially higher values of 4666, 2800, 1800 and 2050 μmol g^{-1} are reported for xylene adsorption, respectively, for activated carbon AC40 (1300 m²/g)(Benkhedda et al, 2000) and zeolithes (Al-Meso 100 (915 m²/g) UL-ZSM5-100-2 (840 m²/g), UL-ZSM5-100-6 (780 m²/g) (Huang et al, 2000)

The numerical integration of the desorption curve (part B of Figure 3) using equation (2) and considering the curve in the absence of solid, permitted quantification of loosely adsorbed fraction (n_{1ads}) of 370 μmol.g^{-1} released during isothermal desorption.

$$n_{1ads} = \frac{FC_{in}}{m}\left[\int_{t_a}^{t_b} \frac{C_{out}}{C_{in}} dt \right] \tag{2}$$

Where t_a and t_b correspond to starting and ending time of isothermal desorption.

It is to be noted that although the o-xylene desorption curve reached zero, the calculated weakly adsorbed fraction represents 88% of the total adsorbed amount. The remaining o-xylene adsorbed amount corresponds to the more strongly adsorbed fraction. The removal of the latter fraction requires thermal treatment with linear heating rate according to TPD method. The integration of the corresponding curve, shown in part C of Figure 3 using equation (3), permits obtaining an irreversible amount of 60 μmol.g^{-1}.

$$n_{2ads} = \frac{FC_{in}}{m} \cdot \left[\int_{t_b}^{tc} \frac{C_{out}}{C_{in}} dt \right] \tag{3}$$

(where t_b and t_c correspond to the starting and the ending of TPD curve).

Therefore, the data corresponding to total adsorbed amount (n_{ads}) were found to fit the mass balance equation ($n_{ads} \approx n_{1ads} + n_{2ads}$) with a precision around 2%, corresponding to the accuracy of the used analytical methodology. It is to be noted that the n_{ads} values do not suffer major changes during successive cycles of adsorption desorption experiments carried out with the same benonite samples and the precision remains at worst around 5%. However, for bentonite sample used for more than 3 successive cycles of adsorption/desorption experiments, a treatment under N_2 flow at 473 K for 30 min, is enough for recovering its initial performances.

Another important information given by TPD experiment concerns the desorption performance indicated by the temperature at the peak maximum. The T_m value of 353 K shown by Figure 3, is slightly, lower than the T_m value of 368 K given by TPD peak obtained using the same heating rate for SiO_2 Degussa (200 m^2g^{-1})(Zaitan et al 2005).

The obtained results reveals adsorptive properties of bentonite clay such as larger reversibly adsorbed fraction, lower temperature for complete thermal desorption and absence of catalytic activity that might be of interest for adsorbent regeneration and adsorbate recovery. Even though its lower BET surface area, Bentonite's potential use as adsorbent material deserves to be investigated because adsorbent with higher specific surface area; such as active carbons is not usually the best (Brasseur et al, 2004). Thus, investigation of adsorption energies is also needed for selection of efficient adsorbent material, particularly, if collecting and reusing VOCs is targeted.

2.4 Isosteric heat of adsorption

The amounts adsorbed at equilibrium corresponding to adsorption capacity obtained from breakthrough curves, were determined for different adsorption temperatures and for different xylene pressures, yields to isotherms shown in Figure 4. The isotherms are represented in the form of $N = f(P)$ where N is the adsorbed amount per adsorbent weight

at equilibrium and P the O-xylene partial pressure in the mixture flow. The experimental isotherms were modeled with Langmuir and Freundlich equations using a nonlinear regression method (MathCAD software). The corresponding fitting curves are shown as solid and dashed lines, respectively, for Langmuir and Freundlich models (Fig. 4). Apparently, the experimental data were well represented by Langmuir model while Freundlich equation deviates at pressures higher than 4 torr.

The adsorption isotherms presented in Figure 4 were used for determination of isosteric heat (Q_{st}) of adsorption by extrapolation at different temperatures and for a given coverage according to Clausius-Clapeyron equation (Rouquerol, 1999):

$$Q_{st} = -R\left(\frac{\partial \ln P}{\partial \left(1/T\right)}\right) \qquad (4)$$

Where R is the perfect gas constant, P and T correspond, respectively; to partial pressure and temperature at equilibrium.
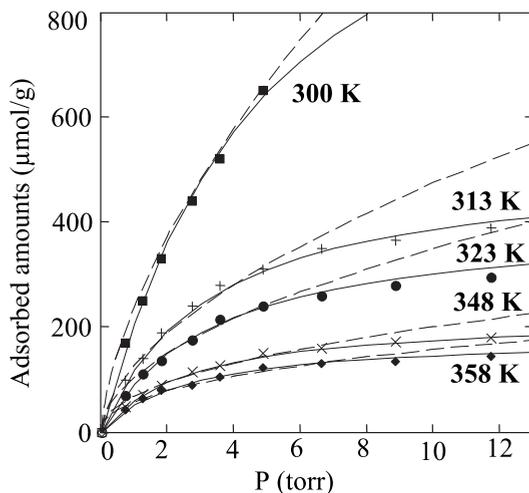
____Langmuir equation
------- Freundlich model

Fig. 4. Experimental and Modeled adsorption isotherms of O-xylene at different temperatures

This approach do not requires an assumption on model fitting with experimental data even though we have shown that Langmuir equation described well the adsorption process in the studied T, P ranges. Thus, (Q_{st}) values were extracted from the slops of isosteres (Figure 5) representing the plot of $\ln(P) = f\left(\dfrac{1}{T}\right)$. One has to check very carefully whether the resulting
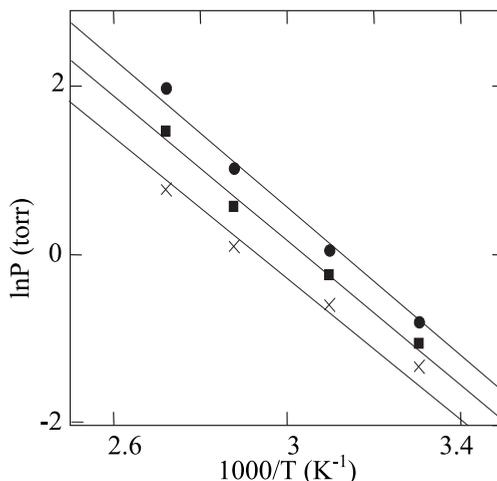
●, 206; ■, 133; ×, 100 µmol g^{-1}

Fig. 5. Plot of isostere corresponding to ln(P) versus1/T corresponding to the following adsorbed amounts

isosteres correspond to straight lines, which depends strongly on the accuracy of experimental conditions. So far, a temperature measurement error of ± 2 K may yield to an uncertainty of ± 8 kJ/mol for the isosteric heat calculation (Bülow et al, 2002 and Dulaurent et al 2000).

For temperatures ranging from 300 to 363 K, values of isosteric heat of adsorption in range of 40 to 44 kJ/mol were obtained for o-xylene loading between 100 and 250 μmol/g corresponding to a coverage between $0.05 \leq \theta \leq 0.125$ ($\theta = n_{ads}/n_{ads\,m}$, where n_{ads} is the adsorbed amount and $n_{ads\,m}$ is the adsorbed amount at monolayer). Nevertheless, these values do not exceed the heat of vaporization (55 kJ/mole) (CRC Handbook, 1985) indicating a weak physisorption adsorption.

2.5 Estimation of the desorption energy with TPD method

In the following section, the part C of the cycle (Figure 3), corresponding to TPD experiment carried out after adsorbent saturation followed by isothermal desorption, will be used for the determination of the desorption energy. The TPD method proposed by Cvetanovic and Amenomiya (Cvetanovic et al., 1967) was also validated under various experimental conditions by several authors (Yang et al.1999; Joly et al.2000; Kanervo et al.2006, Yoshimoto et al.2007). The method allows an easier estimation of the binding energy between the adsorbate and the adsorbent by plotting the desorption rate of the adsorbate as function of temperature. Although, a criticism is reported concerning the assumption related with experimental conditions permitting to achieve TPD measurement free of the influence of diffusion and readsorption (Gorte et al.1996).

The analysis of TPD curves collected at different linear heating rates (β), is based on exploitation of the shift of temperature at desorption peak maximum (T_m) as a function of β. Hence the activation energy for desorption E_d is extracted from the slope of the line obtained from the following equation:

$$2\ln T_m - \ln\beta = E_d/RT_m + \text{constant} \qquad (5)$$

Where T_m is the desorption temperature at peak maximum (K), β is the linear rate of temperature rise (K/min), E_d is the desorption energy (kJ/mol) and R is the perfect gas constant (kJ/molK).

Therefore, in order to provide reliable data on the desorption energy, the TPD curves have to be well defined, with clearly detectable T_m positions. This was the case of our experiment as shown in part C of Figure 3.

Table 1 gives the T_m positions obtained with different heating rates β during the TPD (part C of the cycle). For this purpose additional experiments set of adsorption and desorption cycles at 300K were carried out with the same adsorption pressure of 2.72 Torr of O-xylene (molar concentration of 0.36% in N_2) but with the TPD experiment performed using different linear heating rate β (curves not shown).

B (K/min)	4	5	7	10	12
T_m (K)	350	353	356	361	364

Table 1. Linear heating rate β used for TPD experiments and its corresponding Temperature at Peak maximum T_m (K)

As observed in Figure 6, a linear and positive relationship was observed between $2\ln T_m$ – ln β and $1/T_m$, and a value for the heat of desorption of 75 kJ/mol is obtained from the slope of this plot. This value is higher than those obtained using isosteric method. As stated above, the analytical procedure adopted in the present work permitted quantification of weakly and strongly adsorbed fractions occurring within the predisposed porosity of the clay. Moreover, previous work on textural characterisation revealed that the studied clay is mainly mesoporous and presents wider pore sizes distribution with a significant contribution of pores widths around 9 nm and 40nm (Harti et al, 2007). It is known that the efficiency of an adsorbent depends on its pore structure and the size of adsorbate molecules which affect their diffusion and interaction within the pores. Thus, adsorption in larger pores is associated with lower heats of adsorption corresponding to the weakly adsorbed fraction (n_{1ads}) which represents about 88% of the total adsorbed. While the remaining adsorbed fraction corresponds to the more strongly adsorbed physisorbed species trapped in deeper potentials of predisposed micropores and narrowest mesopores. The resulting heat of adsorption values might be enhanced by "confinement effect", particularly, in case of physisorption of molecules disposing with size similar to those of adsorbent pores (Yoshimoto et al.2007). As shown in the present work, these species will need supplying thermal energy for their transfer to the gas phase, corresponding to the desorption energy which is equal to the heat of adsorption in case of physisorption.
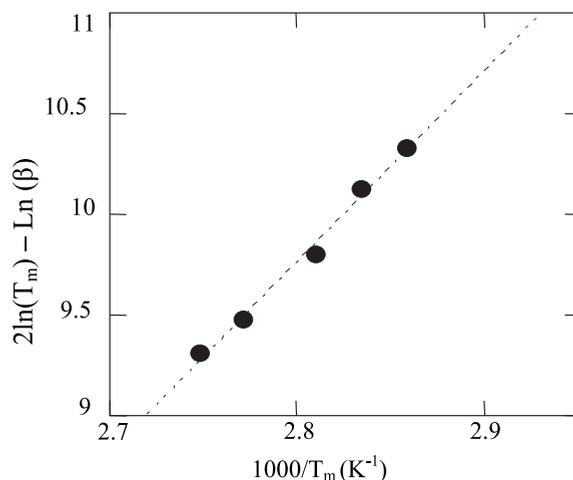

Fig. 6. Cvetanovic curve obtained with TPD experiments performed with β and T_m values presented in table 2 for an adsorption test performed with a flow containing 3600 ppmv of xylene

On the other hand, it is to be noted that the use of Clausius Clapeyron for the estimation of the heat of adsorption, is efficient and accurate when the adsorption process is essentially reversible. This approach is no longer valid for stronger adsorption. As shown in the present work, significant difference might be obtained in the values of the heat of physisorption depending on the nature of the involved porosity. Thus, the analytical method must be able to quantify different adsorbed fractions (i.e. weakly and strongly pysisorption) or else, at best, only an average of the heats of adsorption is obtained, as it is the case of integral or

differential heat of adsorption provided by microcalorimetric methods (Simonot-Grange et al, 1997, Cardona-Martinez and Dumesic, 1997).

3. Conclusion

The use of FTIR spectroscopy was found to permit accurate quantitative analysis that allows determination of adsorbent saturation loading from breakthrough curves and differentiation between weakly and strongly physisorption. The experimental methodology allows, also, the possibility of simultaneous detection of further adsorbate dissociation through appearance of new IR bands. The data obtained using this analytical approach have been used to derivate useful thermodynamic parameters related to the heat involved during adsorption and/or desorption processes. It was shown that significant difference might be obtained for the values of the heat of adsorption depending on to the nature of the involved porosity. This aspect need to be considered for designing and operating of adsorption facilities.

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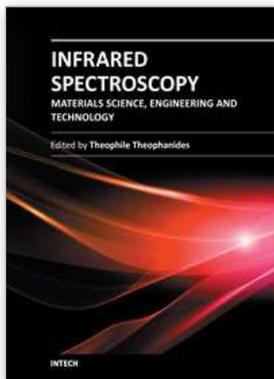
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The present book is a definitive review in the field of Infrared (IR) and Near Infrared (NIR) Spectroscopies, which are powerful, non invasive imaging techniques. This book brings together multidisciplinary chapters written by leading authorities in the area. The book provides a thorough overview of progress in the field of applications of IR and NIR spectroscopy in Materials Science, Engineering and Technology. Through a presentation of diverse applications, this book aims at bridging various disciplines and provides a platform for collaborations among scientists.

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