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Dynamical studies of zeolitic protons and interactions with adsorbates by picosecond infrared spectroscopy

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Abstract

We have investigated zeolite–adsorbate interactions with time-resolved (picosecond) non-linear infrared spectroscopy. In these experiments, the zeolite hydroxyl (the catalytically active, acid site) is vibrationally excited after which the energy relaxation can be monitored real-time. The effect of the presence of adsorbates on energy relaxation rates and pathways provides information on catalyst–adsorbate interaction dynamics. Furthermore, the effect of the presence of adsorbates on the homogeneous linewidth, investigated with infrared hole-burning spectroscopy, provides information on the nature of the interaction potential. © 1997 Elsevier Science B.V.

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1. Introduction

Zeolites, also known as molecular sieves, are crystalline alumino-silicates with large internal surfaces due to the presence of microscopic channels and pores. A large variety of zeolite structures exists \cite{1,2}. The structure of the zeolite known as Y is depicted in Fig. 1. The three-dimensional structure is formed by the interconnecting \( \text{SiO}_2 \) and \( \text{AlO}_2^- \) units; the silicon and aluminum atoms are all tetrahe-
carbon cracking processes, in which long-chain carbon molecules (distillates from crude oil) are converted into more valuable fuel fractions (gasoline and diesel) of smaller molecular size. The cleavage of a C–C bond involves the protonation of one of the carbon atoms, and is therefore an acid-catalyzed reaction; the proton is ‘lent’ by the acid catalyst.

Obviously, the first step in the catalytic conversion at the Brønsted site is adsorption of reactants to the site. Knowledge of the interaction between Brønsted hydroxyls and adsorbed species is therefore essential for a fundamental understanding of zeolite catalysis. Time-resolved infrared spectroscopy has proven to be a powerful tool in the investigation of the vibrational dynamics of the zeolite hydroxyl [4–18]. This paper reports on a time-resolved infrared investigation of the effect of simple adsorbates on the dynamic properties of the catalytic Brønsted hydroxyl. We will partition these effects into elastic and inelastic contributions. By inelastic effects, we denote the influence of the adsorbate on energy transfer rates and pathways at the catalytic Brønsted site. In particular, we will discuss the effect of the adsorbate on the energy relaxation of the vibrationally excited zeolite hydroxyl. This type of information is valuable, since the redistribution of vibrational energy is an important factor in chemical reaction kinetics. In contrast, elastic effects involve adiabatic processes, in which no energy is transferred between different degrees of freedom. It will be shown below that elastic interactions between the hydroxyl and the adsorbate determines the (homogeneous) absorption linewidth of the O–H stretch vibration of the zeolite hydroxyl, because these interactions lead to small frequency fluctuations of the vibrational transition of the hydroxyl. Hence, the homogeneous linewidht also contains information on the zeolite–adsorbate interaction, in particular on the nature of the interaction potential. It is demonstrated that this type of information cannot be obtained with conventional, linear spectroscopy.

2. Experimental

2.1. Zeolite samples

The zeolite samples consist of pressed self-supporting crystalline zeolite discs of \( \sim 5 \text{ mg/cm}^2 \). Acid forms of Y-zeolite and mordenite were obtained by in vacuo heating (1 h at 743 K) of the zeolite in which (part of the) Na\(^+\) cations were exchanged by NH\(_4\) cations. The two types of Y zeolite used in this study are specified by Si/Al-ratios of 2.8 and H/(Si + Al)-ratios of 0.27 and 0.07, respectively. For the mordenite the Si/Al- and H/(Si + Al)-ratios are 6.7 and 0.13, respectively. In this study, we investigate deuterated zeolite samples [19], because of the three times smaller bandwidth (allowing for better spectral resolution) and a reduced scattering of the infrared light at O-D stretch frequencies compared to O-H frequencies. Deuteration is achieved by exposing the zeolite disc to 500 mbar of D\(_2\)-gas (Messer Griesheim, 99.7\%) at 693 K and allowing exchange for 1 h, resulting in approximately 70% exchange as observed from the absorption spectra. Adsorption of nitrogen (N\(_2\), Messer Griesheim, 5.0), xenon (Xe, Messer Griesheim, 4.0), methane (CH\(_4\), L’Air Liquide, 3.5) and carbon-monoxide (CO, Linde, 3.7) was performed at pressures ranging from 50 to 200 mbar, at 100 K.

2.2. Time-resolved non-linear infrared spectroscopy

For the infrared experiments, ultrashort (25 ps), powerful infrared laser pulses are obtained by means of parametric infrared generation and amplification in LiNbO\(_3\) crystals. The 1064 nm (9400 cm\(^{-1}\)) pulses from an actively and passively mode-locked Nd:YAG laser (repetition rate 10 Hz) are used to pump two identical parametric branches, resulting in
energy per pulse for the generated infrared light. The generated pair of laser pulses (pump and probe), independently tunable in wavelength from 2200 to 7200 cm$^{-1}$, are used for so-called pump-probe experiments. In these experiments, the effect of a powerful pump ($\sim 150 \mu$J) pulse are monitored by a weak probe pulse ($\sim 3 \mu$J), as a function of (i) time delay between pump and probe and (ii) frequency of the probe pulse: Transient spectra can be recorded as a function of delay time [11].

In the experiments, the frequency of the pump pulse is tuned to frequency of the vibration under investigation, e.g. the O–D stretch vibration ($\nu_{\text{OD}} \approx 2600 \text{ cm}^{-1}$) of the deuterated zeolite hydroxyl. The non-linear character of this spectroscopic technique lies in the fact that this pump pulse excites a significant fraction (10\%) of the O–D groups from their vibrational ground ($\nu = 0$) state to their first vibrationally excited ($\nu = 1$) state. For the duration of the excitation (the excited state population lifetime), these O–D groups can no longer absorb the infrared probe light at the transition frequency, since they are already excited to $\nu = 1$. Thus, it is clear that the pump-induced changes in the infrared absorption spectrum can be investigated by monitoring the transmission of the (much weaker) probe pulse. The transmission decay back to equilibrium reflects decay of the excited state population and can be followed real-time, since the probe pulse can be variably delayed with respect to the pump pulse. Decay of the excitation, viz. vibrational population (or energy-, $T_1$-) relaxation, is mostly non-radiative and entails the redistribution of the vibrational energy into a set of modes with lower energy which are anharmonically coupled to the excited mode [20–22]. These modes are called ‘accepting modes’: together these receive the excess energy upon de-excitation of the oscillator.

3. Inelastic effects of the adsorbate

3.1. Enhancement of the relaxation rate by adsorbates

A downshift of the O–D absorption frequency occurs upon adsorption of argon, nitrogen, oxygen and methane on the deuterated zeolite mordenite at $100 \text{ K}$, as depicted in the upper panel of Fig. 2. This is caused by a weakening of the original O–D bond due to the presence of the hydrogen bond between the hydroxyl deuteron and the adsorbed species [23]. Note that upon adsorption of N$_2$ and CH$_4$ two distinctive peaks appear: a low-frequency peak due to the perturbed species, i.e. hydroxyls at which adsorption is occurring, and the residue of the non-perturbed species, on which no adsorption takes place, at higher frequencies.

Two typical results of pump–probe experiments for the case of nitrogen adsorption are depicted in Fig. 3. The absorption spectrum for this sample is shown as the dotted line in the upper panel of Fig. 2.
3.2. The effect of the adsorbate on the relaxation rate

The rate of vibrational relaxation $k_{vr}$ of the excited vibration is generally described by Fermi’s golden rule [20,21,24]:

$$k_{vr} = \frac{2\pi}{\hbar} \sum_{a,a'} |\langle r', a' | V(r, a) | r, a \rangle|^2 \times \rho(E) \delta(E(r', a') - E(r, a)).$$

In Eq. (1), $r$ and $r'$ designate the initial and final state of the initially excited vibration (the O-D stretching mode, in our case), while $a$ and $a'$ refer to the accepting modes. The ket wavefunction $|r, a\rangle$ denotes the system with excited O-D stretching mode and thermally populated accepting modes. The bra wavefunction $\langle r', a'|$ denotes the final state, after relaxation. The density of states is $\rho(E)$ for the accepting modes in the relaxation process and the delta-function $\delta(E(r', a') - E(r, a))$ ensures conservation of energy. The summation shows that the true relaxation rate is a sum of contributions from all possible pathways. The increase in relaxation rate by the hydrogen bond with the adsorbate can therefore be caused by (i) a change in the potential $V$ (possibly accompanied by changes in the accepting mode density of states $\rho(E)$, when new channels become available) or (ii) an increase in overlap between the final $\langle r', a'|$ and initial $|r, a\rangle$ wavefunctions. In the following, we will discuss the possible contributions of these two factors to the increased rate.

The presence of the hydrogen bond with the adsorbate creates new possible accepting modes in which (part of) the energy can be dissipated. In particular, it could be that the internal low-frequency modes of the adsorbed molecule act as accepting modes, allowing for part of the excess vibrational energy to be transferred to an adsorbate vibrational mode. This means that, upon adsorption, in the anharmonic coupling term $V$ contributions of the form $R^p_{OD} \cdot R^q_{Adv}$ (with $p, q > 0$) must be considered, so that energy from the excited O-D stretching mode (designated by coordinate $R^p_{OD}$) can flow into the adsorbate (designated by coordinate $R^q_{Adv}$). In this case, one would expect the relaxation rate to depend critically on the exact internal low-frequency
mode distribution of the adsorbed species. We observe, however, that the lifetime is hardly dependent on the adsorbed species (Fig. 2, only for Ar the lifetimes seem somewhat larger). This leads us to conclude that there is no direct energy flow from the excited hydroxyl into internal degrees of freedom of the adsorbed molecule: If specific modes of the adsorbed molecules would act as accepting modes, one would expect different lifetimes for different adsorbates, contrary to what is observed.

A second possible new relaxation channel is the hydrogen bond itself: One might expect that, since the hydrogen bond lies along the O–D stretch vibrational coordinate, energy transfer from the excited O–D group into the hydrogen bond would be very efficient. In fact, it has been observed that the breaking of hydrogen bonds is a very effective relaxation channel for vibrationally excited hydrogen-bonded hydroxyls [12,25]. In this case, the anharmonic coupling term V contains contributions of the form \( R_{OD}^p \cdot R_{H\_bond}^q \) (with \( p, q > 0 \)), so that energy from the excited O–D stretching mode (designated by coordinate \( R_{OD} \)) can flow into the hydrogen bond (designated by coordinate \( R_{H\_bond} \)). In this case, the hydrogen bond acts as the primary accepting mode, thus accelerating the relaxation process [13]. For the zeolite, this would result in a one-photon desorption process of the adsorbate upon vibrational relaxation of the hydroxyl. The heat of adsorption of the adsorbates considered here is typically 1000 cm\(^{-1}\)/bond, i.e. about one third of the vibrational quantum [26]: There is sufficient energy in the excited hydroxyl to dissociate the hydrogen bond. Indeed, we observe desorption of nitrogen from the zeolite hydroxyl. This is demonstrated by the transmission increase after vibrational relaxation, which means that OD...N\(_2\) groups have been converted to bare OD groups with a different frequency. However, experiments with decreasing pump energy revealed that the desorption is indirect, caused by a rapid heating of the zeolite lattice due to the laser pulse [8,9]. Hence, in contrast to other hydrogen-bonded systems, direct dissociation of the hydrogen bond as a one-photon desorption process, does not occur upon relaxation of the vibrationally excited zeolite hydroxyl. Although minor energy flow is still a possibility, this rules out the possibility of the hydrogen bond as the primary relaxation channel. It was shown recently that the picture that the hydrogen bond acts as an additional accepting mode (in contrast to a primary accepting mode) is consistent with the temperature dependence of both the O–D and O–H vibrational lifetimes [13].

The possible effect on the wavefunction overlap can be partitioned into effects on the \( r \)-coordinates and on the \( a \)-coordinates. By increasing the (self-)anharmonicity of the O–D stretching mode (\( r \)-coordinate) the overlap will evidently increase [7], resulting in a faster decay. On the other hand, the accepting modes (\( a \)-coordinates) will also be affected by the hydrogen bond: The energy spread of the accepting modes (as well as the width of the absorption band of the O–D stretching mode itself) could be broadened by the hydrogen bond [27]. This will result in an increased overlap between the \( |r, a\rangle \) and \( \langle r', a'| \) wavefunctions, at the same energy. In this case, the enhanced relaxation rate can be interpreted as an energy mismatch compensation by the hydrogen bond [27]: The hydrogen absorbs a very small part of the excess vibrational energy, thus, ensuring a better energy match, and a faster relaxation. An increase in the wavefunction overlap due to interaction with the hydrogen bond requires that the hydrogen bond is different for the ground and excited state of the O–D stretch vibration. This could be caused by a different H-bond strength upon excitation of the O–D stretch vibration.

It was proposed recently [18], that the increased relaxation rate is caused by the detuning of the hydroxyl stretch vibration. The detuning results in a better energy match between the O–D stretch energy and the sum of energies of the accepting modes plus one or two phonon modes. However, this explanation seems not very likely, since the density of states of the low-frequency modes is very broad [13,28–30], and the 3, 4 or 5-fold compound density of states, relevant for the relaxation process will be even broader.

Summarizing, we observe a dramatic effect of the hydrogen bond on the relaxation rate. We find no evidence for direct energy transfer between the catalytic hydroxyl and the internal degrees of freedom of the adsorbate. The bulk of the excess energy of the vibrationally excited hydroxyl is still dissipated over the zeolite lattice. The enhanced relaxation is therefore attributed to an increase in overlap between the wavefunctions before and after relaxation, where
we cannot differentiate between different contributions from the two coordinates, and the possibility that the hydrogen bond acts as an additional accepting mode.

4. Elastic effects of the adsorbate

4.1. Infrared holeburning studies

The width of infrared absorption lines of condensed phase matter is generally determined by inhomogeneous broadening [31]: The different groups in the sample all have slightly different transition frequencies due to impurities, defects or other inhomogeneities. This means that the width of the overall absorption line only contains information about this static frequency distribution, generally different from sample to sample. For a sub-group with the same center-frequency, the transition will also exhibit a finite width. In contrast to inhomogeneous broadening, this broadening is caused by dynamical interactions of this sub-group with its surroundings, and is called homogeneous broadening [31]. These interactions can be viewed as adiabatic (i.e. no energy is transferred) interactions: They lead to rapid, small modulations of the vibrational transition frequency.

With non-linear infrared spectroscopy, inhomogeneous and homogeneous contributions to the absorption lines can be separated. In our non-linear pump-probe experiments, the pump pulse is tuned to the top of the absorption band of the zeolite hydroxyl. If the absorption line is strongly inhomogeneously broadened, the pump pulse will 'burn a spectral hole' in the absorption band: only the groups resonant with the excitation pulse can be excited. In case of homogeneous broadening, the whole absorption band will decrease in amplitude upon excitation.

In Fig. 4 two absorption spectra in the O–D stretching region are shown, as recorded with the infrared probe pulses (□’s in upper and lower panel). The upper panel shows the absorption spectrum for D_{0.19}Y zeolite (36 O–D groups per A_{152}Si_{140}O_{396} unit cell). The absorption peak around 2620 cm\(^{-1}\), originates from the low-frequency (LF) sites in the small cages of this zeolite [32]. These hydroxyls are known to be hydrogen-bonded to zeolite oxygen atoms (e.g. on the other side of the small pore) and will be further referred to as OD...O_{latt} groups. The lower panel depicts the absorption spectrum of the same zeolite with a lower concentration of O–D groups, NaD_{0.05}Y (10 O–D groups per unit cell), after addition of 50 mbar of nitrogen at 100 K. In this zeolite, there are mainly hydroxyls in the large cages (v_{OD} = 2680 cm\(^{-1}\)). The nitrogen adsorbs to these O–D sites, resulting in a redshift of the peak from 2680 to 2625 cm\(^{-1}\) due to the weak hydrogen bond to the adsorbate [26]. The resulting O–D groups will be further referred to as OD...N$_2$ groups. Note the similarity between the absorption bands of the LF O–D peak of D_{0.19}Y due to the OD...O_{latt} groups and that of the HF O–D of NaD_{0.05}Y with adsorbed nitrogen, due to the OD...N$_2$ groups. The

Fig. 4. Regular (□) and transient (●) absorption spectra (recorded with the infrared laser probe pulses) with and without the pump pulse tuned to the peak of the absorption band of two hydroxyls in Y zeolite. For the O–D...O_{latt} groups, the width of the hole is almost completely determined by the 6 cm\(^{-1}\) width of the laser pulses, revealing a strong inhomogeneity. For the O–D...N$_2$ groups, the homogeneous absorption line is over an order of magnitude larger. For both experiments T = 100 K.
vibrational lifetimes of these two differently hydro-
gen-bonded groups are also extremely similar [16],
44 and 48 ps, respectively.

The effect of excitation with the pump-pulse on
the absorption spectra is depicted in the same figure
(●'s in upper and lower panel), where the absorption
lines (recorded with the weak probe pulses) with
and without the pump pulse are presented. In these
experiments, the 6 cm\(^{-1}\) broad pump pulse is tuned
to the peak of the absorption spectrum, the delay
between pump and probe is fixed at 30 ps and the
probe frequency is scanned. It is found that the pump
pulse burns a hole in the absorption spectrum for the
O–D...O\text{lat} groups, whereas the whole absorption
band goes down in amplitude for the O–D...N\text{\textsubscript{2}}
groups. The fact that a hole is burnt in the O–D...
O\text{lat} absorption spectrum implies that this line
is strongly inhomogeneously broadened [33]; there is
a distribution of vibrational frequencies for the O–D
groups, and the laser pulse can only excite those
O–D groups resonant with the frequencies within the
laser pump pulse at the center of the absorption
band. This means there is an inhomogeneous distri-
bution of catalytic sites in acid zeolites [11]. A
possible cause for the inhomogeneous broadening is
the inhomogeneous distribution of silicon and alu-
mminum atoms throughout the lattice. It is known that
even the second coordination shell of Al/Si atoms
is ‘felt’ by a Si or Al atom [34]. Since we have
observed that within the LF O–D (O–D...O\text{lat})
absorption band the lifetime decreases strongly with
decreasing frequency [14], the inhomogeneous distri-
bution of Al atoms evidently gives rise to a static
distribution of hydrogen bond strengths: More
strongly hydrogen-bonded O–D groups will absorb
at lower frequencies, and exhibit shorter lifetimes.
For the O–D groups with adsorbed nitrogen, the
homogeneous absorption linewidth is over an order
of magnitude larger than the homogeneous linewidth
in vacuum.

4.2. The zeolite–adsorbate interaction potential

The behaviour of the homogeneous linewidth for
different adsorbates [15,16], leads to the schematic
hydrogen-bond potentials drawn in Fig. 5. Shown in
Fig. 5 is the variation of the energy of the system
with the hydrogen-bond coordinate. For the OD
groups in vacuum (hydrogen-bonded to the zeolite
lattice) this coordinate is the O\text{D}...O\text{lat} distance, for
the OD groups with e.g. adsorbed nitrogen, it is the
O\text{D}...N\text{\textsubscript{2}} distance. In the presence of nitrogen, the
potential is strongly anharmonic. In our experiments,
we excite the O–D stretch vibration to its first
excited state (arrows pointing up in Fig. 5 designate
such a transition; the presence of the H-bond lowers
the frequency from ~ 2750 to ~ 2600 cm\(^{-1}\)). It is
well known that the excited (\nu_{\text{OD-stretch}} = 1) state
potential is different from the ground (\nu_{\text{OD-stretch}} = 0)
state potential [35]; the hydrogen bond is stronger for
the \nu = 1 excited state than for the \nu = 0 ground
state, and hence the potential energy minimum is
situated at smaller O...N\text{\textsubscript{2}} distance for \nu = 1 [35].
This means that, for the O–D groups with adsor-
bates, a variation of the O...N\text{\textsubscript{2}} distance will result
in a change (modulation) of the \nu = 0 \rightarrow \nu = 1 O–D
stretch transition frequency. In other words, the exact
transition frequency for the O–D group depends on
the ON\text{\textsubscript{2}} distance. This culminates in very broad
absorption lines for the individual hydroxyls and
explains the fact that it is relatively difficult to
differentiate the hydroxyl groups spectroscopically.
In contrast, for the OD...O\text{lat} groups the potential energy curves are dictated by lattice parameters; the OD...O\text{lat} distance is not determined by the electrostatic interaction between the hydrogen and the O\text{lat}, but by the geometry of the zeolite lattice. To move the O\text{lat} away from the O–D group requires a local deformation of the zeolite lattice, energetically very unfavorable. Hence the potential is very narrow and harmonic, and very similar for the ground and excited state of the O–D vibration. The fact that the ground and excited state potentials are the same implies that, in contrast to the O–D groups with adsorbates, the transition frequency of the O–D stretching mode does not depend on the OD...O\text{lat} distance: the presence of this type of hydrogen bond does not affect the absorption line of a sub-ensemble of O–D groups with the same center frequency. Indeed, the homogeneous absorption lines are much narrower for the OD...O\text{lat} groups than for the O–D groups with adsorbates.

In summary, we see that the homogeneous linewidth contains information on the zeolite–adsorbate interaction potential. It should be noted that the homogeneous linewidth can only be obtained with non-linear optical techniques, such as the spectral hole-burning technique presented here.

5. Conclusions

Picosecond non-linear infrared spectroscopy applied to zeolites, provides information on the interaction dynamics between the zeolite catalyst and adsorbates, along with structural information on the catalytic sites. As far as energy transfer is concerned, we find no evidence for energy transfer processes between the zeolite hydroxyl and the adsorbate and observe that the hydrogen bond between the hydroxyl and the adsorbate is not directly dissociated. We conclude that the enhancement of the vibrational relaxation rate is due to an increase in the coupling between the hydroxyl and the zeolite lattice, presumably caused by a minor energy flow into the hydrogen bond, which acts as an additional accepting mode.

Furthermore, we have examined the homogeneous linewidths of the O–D stretching bands of the zeolite hydroxyl with infrared hole-burning spectroscopy. In vacuum, we observe a strong inhomogeneous distribution of hydroxyls, which decreases upon adsorption of simple molecules such as nitrogen. It is demonstrated how the elastic interactions determining the linewidth are connected to the hydroxyl–adsorbate interaction potential.

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From the experiments that were performed on the non-deuterated O-H groups, we deduce that the results for the O-D groups hold for the O-H groups as well.