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Operando IR of catalytic reactions under microwaves at 5.8 GHz

Frédéric Thibault-Starzyk^{a,*}, Ana Neto^{a,b}, Sébastien Thomas^{a,1}, Carlos Henriques^b, Gary Bond^c

^a Université de Caen Normandie, ENSICAEN, CNRS, LCS, Caen 14000, France

^b Centro de Química Estrutural, Instituto Superior Técnico, Universidade de Lisboa, Av. Rovisco Pais, Lisboa 1049-001, Portugal

^c School of Engineering and Computing, University of Central Lancashire, Preston PR1 2HE, United Kingdom

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ABSTRACT

A new spectroscopic cell was designed for operando IR observation of heterogeneous catalysis under microwave irradiation. Oxidation of CO on Pt-H-BEA zeolite was performed under conventional and microwave heating at the same space velocity to assess the efficiency of microwave heating, which was confirmed by an infrared in-situ temperature scale. A temperature of 230–240°C was obtained after 2 min microwave irradiation. Infrared monitoring evidences a moderate sintering of the platinum particles after microwave irradiation, probably due to hotspots on the metal. Infrared observation indicates a strong perturbation of electron density in the Pt particles by microwave during catalysis.

1. Introduction

Microwave (MW) heating has been known for some time for its benefits in organic or inorganic syntheses [1,2]. More recently, the use of microwave heating in heterogeneous catalysis has attracted much interest [3–5], with several publications reporting an enhancement of the reaction yields, changes in selectivity, as well as reduction of the bulk reaction temperature compared to conventional heating. Possible explanations were attributed to the creation of different temperature regimes in the reaction system, with the formation of hot-spots or specific heating of metal nanoparticles by microwaves [6,7] or to athermal effects (mode-selective excitations) in the case of molecules adsorption in the micropore system of zeolites [8].

Several researchers point to MW catalysis as a field-responsive domain driven by localized energy deposition and proposed unique advanced spectroscopic tools [9,10].

Infrared (IR) operando spectroscopy is a very powerful technique for the study of the dynamic behaviour of catalytic systems under real working conditions [11,12], but the design and fabrication of a combined IR-MW cell needs addressing several engineering constraints. The first obvious need is IR transparency of both the cavity and the sample while preserving homogeneity of the MW field. IR transparency is usually achieved with a very small sample, ideally a 10–20 mg very thin self-supporting wafer. Temperature stability (with protection of gaskets

and windows) is also needed, since heterogeneous catalysis is often controlled and tuned by heating. Previous attempts used microwave applicators inside the IR reactor-cell, connected to the MW generator via coaxial cables [13,14]. Connection of the coaxial cables with the miniaturized applicator plates and insulation from the body of the cell were often difficult, especially at variable temperature, which rendered these designs difficult to master.

In the present work, a new reaction cell was developed with simple waveguides for coupling microwave heating with operando IR analysis. This design is easy to use and allows time resolved studies of transient surface chemistry, giving new insights into the unique influence of microwaves on reaction mechanisms and selectivities.

2. Experimental part

2.1. Development of the microwave reactor – IR cell

A microwave cavity was designed for *in situ* IR spectroscopy studies under microwave irradiation at 5.8 GHz (Fig. 1 A and B).

The sample used in transmission IR operando spectroscopy is a very small self-supported wafer (20 mg, 1.3 cm², 50µm approximate thickness). This unusual sample geometry and the need for cavity miniaturization strongly influenced the choice of microwave frequency and the design of the cavity.

* Corresponding author.

E-mail address: fts@cnrs.fr (F. Thibault-Starzyk).

¹ Present address: ICPEES, CNRS-University of Strasbourg, 25 rue Becquerel, Strasbourg 67087Cedex 02, France

The volume of the workload can be reduced as the frequency increases, which becomes advantageous when trying to heat a small self-supported wafer [15]. Microwave at 5.8 GHz frequency was used rather than 2.45 GHz to improve the power density dissipated as heat in such a small sample volume.

The cavity was made from a standard rectangular waveguide with a reduced-height section and two tapered sections (*surfaguide* design). The tapered sections ensure a smooth transition from the standard waveguide to the reduced-height waveguide. This type of waveguide has been used to allow the intensification of the electric field in the design of plasma generators [15]. In the present case, plasma production is not desired but an intense microwave field is needed to maximize the microwave energy and effectively heat the very small self-supported wafer. The modified waveguide was constructed in copper (250°C maximum operating temperature).

Observation ports allow infrared observation through the cavity. A glass tube goes through the observation ports and the cavity, with gas inlet and outlet on each side and CaF₂ windows. The glass tube also plays the role of the sample holder inside the waveguide so that the catalyst wafer (ø 13 mm) is positioned in the middle of the microwave cavity and perpendicular to the IR beam.

The setup is shown in Fig. 1.

A directional coupler (50 dB, WR159, with min directivity 27 dB) is installed after the microwave cavity for power monitoring during experiments. The coupler is connected to a power sensor (E-probe, Agilent Technologies, E4413A) and the power measurement acquisition is performed using a wattmeter from Agilent Technologies (E4418A).

Tuning of the microwave cavity is checked using the power monitoring system, so that the maximum of the E-field is located at the wafer position. For this reason, the power sensor was placed at multiple of $\lambda/2$ from the sample position. The plungetuner (supplied by ARVA-RF) is used for tuning of the cavity, as well as a 3-stub-tuner (ARVA-RF, SWR of 1.25:1) used for fine-tuning. The microwave source is a variable power (150 – 1000 W) 5.8 GHz magnetron generator provided by Covimag (France). Since the sample is only 20 mg, absorbed power is a small fraction and most power is reflected to the water load.

In order to combine conventional with MW heating, a heating coil was wrapped around the cavity to form a conventional oven and perform the experiment at the desired temperature. The maximum

operating temperature for the modified waveguide is 250°C to prevent deformation of the waveguide and cavity. Since the heating cable is placed on the outside of the waveguide, the sample will only heat via conduction and the temperature inside the microwave cavity is lower than the temperature of the Thermocoax® cable itself. The sample's temperature inside the IR cell was calibrated (under a flow of argon). A sample was placed inside the IR cell. One of the CaF₂ windows was replaced with a pierced window through which a temperature probe (K thermocouple) was introduced next to the sample. The variation of the temperature reading inside the cell was registered as the temperature set point of the heating cable was changed. A maximum of 170°C was found when the heating wire was set to 240°C.

2.2. Catalyst

The catalyst used was a zeolite BEA containing 1.13 wt% Pt. This sample was prepared using zeolite BEA (ammonium form, Si/Al=12.5, Zeolyst CP814E) and the platinum was introduced using [Pt(NH₃)₄]²⁺ ion exchange (from Pt(NH₃)₄(NO₃)₂, Aldrich). Pt dispersion (64 %, 1.5 nm average particle size) was determined by static CO chemisorption followed by FTIR spectroscopy. The self-supporting wafer (13 mm, 19.8 mg) was prepared under moderate pressure (10⁸ Pa, 1 t.cm⁻²).

Pre-treatment of the sample by conventional heating was performed *in situ* to ensure that all Pt atoms would be reduced. The sample was first heated (5 °C/min up to 120 °C) under argon (30 mL/min) to remove all water from the catalyst surface. After that, hydrogen (30 mL/min) was introduced in the cell, further heated (5 °C/min up to 170 °C) for reduction of the supported platinum in the catalyst.

2.3. CO oxidation

All catalytic tests, under microwave or conventional heating, were performed in the same reactor cell, thus insuring similar flow conditions for all experiments. After pre-treatment, the sample was cooled down to room temperature (microwave experiment) or to 50 °C (conventional heating) under argon flow until the mixture of CO (1 % in Ar, 3.15 mL/min) and O₂ (2 % in Ar, 6.15 mL/min) was fed to the reactor (0.34 % CO and 1.30 % O₂, 9.3 mL/min total flow).

After equilibration of the reactant gas flow inside the reactor, the

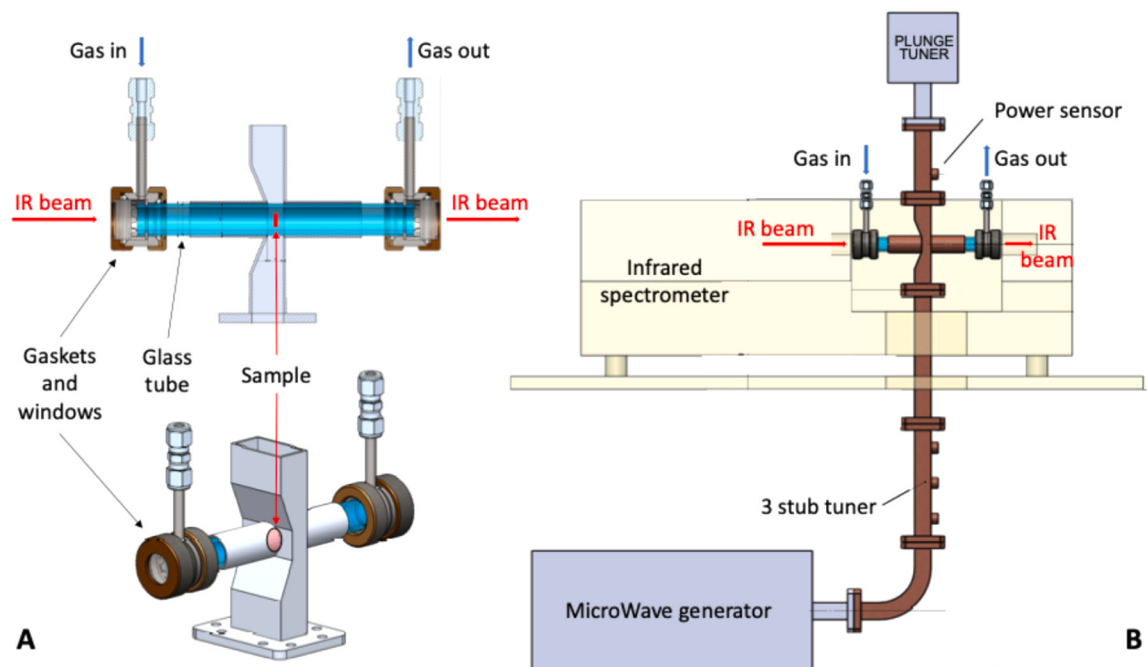


Fig. 1. Schematic representation of the microwave cavity and design of the reactor (A), and schematic of the whole microwave setup for spectroscopic studies (B).

reaction was started by conventional (5 °C/min) or microwave heating. The catalyst surface was monitored with infrared spectroscopy. CO consumption was monitored with an online IR gas cell on the reactor outlet.

The spectrometer used was a Nicolet Magna 750, with an MCT/A detector (velocity 1.8988, 4 cm⁻¹ data resolution, 32 scans per spectrum, 1 spectrum every 13 s).

3. Results and discussion

A simple reaction was chosen to evaluate the heating ability of the system developed: CO oxidation reaction with a bifunctional catalyst of 1 wt% Pt on BEA zeolite. The results were compared with those obtained for the same reaction using conventional heating in the same reactor-cell.

CO oxidation is a common model reaction for heterogeneous catalysis [16–18]. It was used as an *in-situ* temperature probe in microwave heating of γ -alumina supported Pt and Pd catalysts [19,20]. Silverwood et al. also used CO oxidation to study the heating efficiency of a microwave-heated IR cell [13]. In the present study, the temperature at which maximum CO conversion is achieved is compared for both conventional and microwave heating systems.

3.1. Using infrared bands as *in situ* temperature probes

Many infrared bands in oxides shift with temperature. This red-shift is related to the influence of the temperature on the crystalline structure and to the anharmonicity of the vibrations [21]. $\nu(\text{OH})$ vibration bands for zeolite's silanol groups around 3740 cm⁻¹ are particularly clear, with isolated silanols at 3745 cm⁻¹ and internal silanols at crystalline defects at 3737 cm⁻¹ [22]. The $\nu(\text{OH})$ band of isolated silanol groups at 3745 cm⁻¹ shifts linearly with temperature and we used it as a *in situ* probe for the temperature (Fig. 2, left).

The position of the 3745 cm⁻¹ band was determined using the second derivative of the spectra. The relationship with temperature is shown in Fig. 2 (right pane). This relationship was later used to determine the sample's temperature variation under microwave irradiation. It should be noted that the wafer is very thin (probably less than 50 μ). Spectroscopic observation, being done across the wafer, is an average of the physical state (and temperature distribution) over the IR optical path through the wafer.

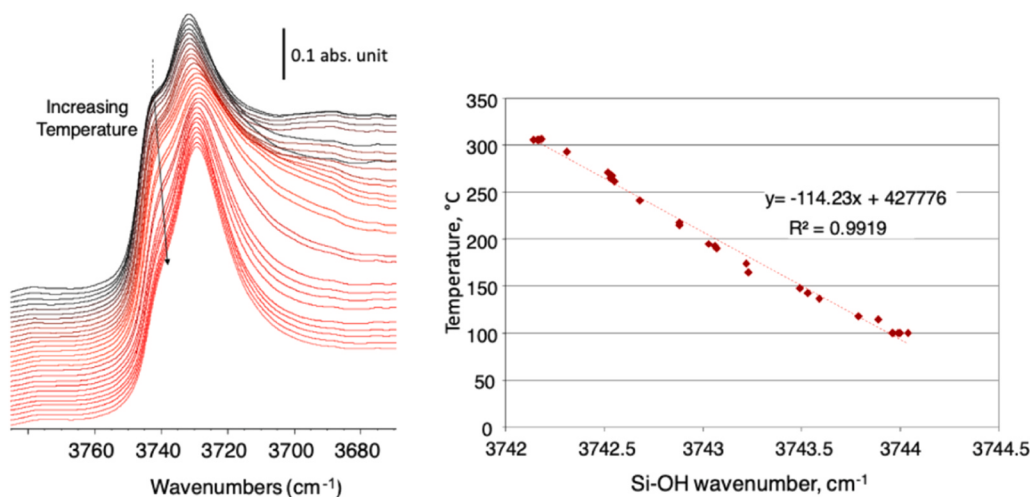


Fig. 2. Left: Red-shift of the 3745 cm⁻¹ band observed in the $\nu(\text{OH})$ region of the sample spectra between 100 °C and 300 °C; Right: relationship between the catalyst temperature and the position of the Si-OH vibration band.

3.2. CO oxidation under conventional heating

The reactor-cell was first tested with conventional heating, without microwave. Although the design of the reactor is very different from that of a typical plug-flow, the comparison between both heating methods is assumed to be valid.

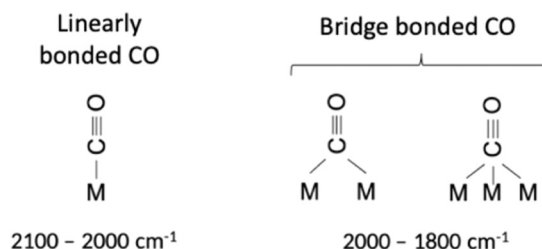
The interaction of carbon monoxide with transition metals normally results in carbonyl complexes [23], which appear in the 2100 – 1800 cm⁻¹ region (Scheme 1).

When using platinum as the metal active phase, linearly bonded CO is usually found. Sometimes, a band due to CO bridge-bonded to two platinum atoms, ca. 1860 cm⁻¹, may also be observed. On the other hand, if non-reduced platinum is present, the spectroscopic data become more complex, with several features appearing above 2100 cm⁻¹ corresponding to platinum in exchange positions, Pt^{δ+}(CO)_n [24].

In the present case, only linearly bonded CO is observed in the sample spectra around 2073 cm⁻¹. The immediate increase of the 2073 cm⁻¹ band after addition of the reactant gas to the operando cell can be seen in Fig. 3A. The bands appearing below 2000 cm⁻¹ are combinations of vibrations of the zeolite structure. As the sample is heated (Fig. 3A, spectra e to i), the CO (interacting with Pt⁰ particles: Pt⁰-CO) band disappears due to oxidation of the carbon monoxide in the system. Complete conversion of the CO is achieved and the 2073 cm⁻¹ is no longer observed at high temperature.

CO consumption was monitored using the IR gas cell. Fig 3B shows the spectrum of the reactor gas effluent while CO is being fed to the system, before heating the catalyst.

The evolution of the area of the CO band in the gas phase, as a function of temperature, gives the CO consumption profile (Fig. 3C). As the reaction takes place, the carbon monoxide disappears from the gas stream to produce carbon dioxide, which is visible by the decrease in



Scheme 1. Various interactions of CO with supported Pt on oxides, and the corresponding $\nu(\text{CO})$ infrared band frequencies.

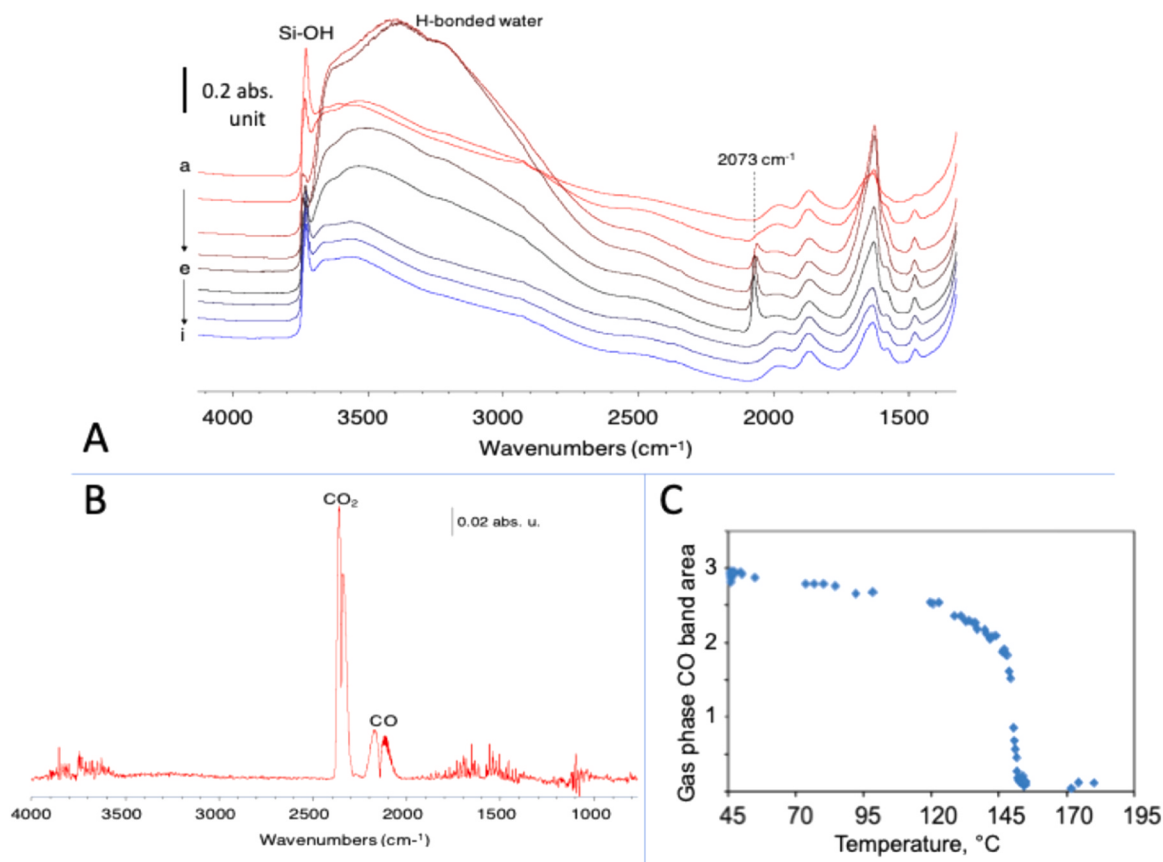


Fig. 3. A: Evolution of the sample spectra (a) at 45 °C before addition of the CO+O₂ mixture; (b – e) during equilibration of the gas flow rate at 45 °C; (e – i) during heating of the sample at 5°C/min (e: 45 °C, f: 80 °C, g: 112 °C, h: 147 °C, i: 180 °C). B: Gas phase spectrum of the reactor effluent, while the reactant gas is being fed to the system (corresponding to spectrum e on the solid phase). C: evolution of the quantity of unreacted CO in the gas phase exiting the reactor.

intensity of the $\nu(\text{CO})$ band, and consequent increase of the $\nu(\text{CO}_2)$ band. A sudden decrease in the CO band intensity is observed for two consecutive spectra (recorded only 13 s apart), around 150°C.

3.3. CO oxidation under microwave Heating

The reactor – IR cell was further tested in the CO oxidation reaction under microwave irradiation, without conventional heating. The changes taking place at the surface of the catalyst were followed by FTIR spectroscopy, as well as the gas products at the reactor outlet.

As mentioned in the introduction, a very small sample is used to achieve IR transparency in the MW reactor-cell (ideally a 10–20 mg very thin self-supporting wafer). It should be noted that microwave heating of oxide or zeolite supported metal catalysts, especially in such small quantities, remains an open question. Three possible heating mechanisms can be proposed:

- Measurements of dielectric properties of zeolite have shown the key role of adsorbed water [25]. In the present experiment, dry materials are used and water is carefully excluded from the system.
- Metal contained in the catalyst could contribute to the heating of the solid. In the present sample, Pt is in very small amount and present in nm size particles. Although local hotspots might appear on these metal particles, heating of the whole sample by this mechanism in the timeframe of our experiment can be ruled out.
- Heating of zeolites could also be due to cation mobility [5], as reported in a previous work on zeolite beta [26]. However, the present zeolite sample was thoroughly exchanged (to the ammonium form and with some ammonium-platinum salt) and further calcined so that it contains no remaining cation (Pt is reduced as Pt⁰), hence

there are no or very few mobile charge carriers in the zeolite catalyst used in the present study.

Rationale of MW heating of a such dry and nonionic zeolite is thus not straightforward, especially with the present sample geometry, and checking the achieved temperature is very important. Several research groups started addressing the question with advanced spectroscopic techniques [10]. The IR in situ scale obtained with the shift of the SiOH vibration band, as presented higher in this paper, was applied here.

The gas products followed the same behaviour during microwave irradiation as under conventional heating of the catalyst. The $\nu(\text{CO})$ band decreased in intensity during the test, while the $\nu(\text{CO}_2)$ became more intense, indicating the conversion of carbon monoxide into carbon dioxide.

The evolution of the sample spectra prior, during and immediately after microwave irradiation is presented in Fig. 4A. Before microwave irradiation, the Pt⁰-CO band can be seen at 2077 cm⁻¹ on the solid, as well as carbon monoxide (and a small amount of carbon dioxide) in the gas phase.

When microwaves were applied, the signal-to-noise ratio immediately dropped and the spectral quality was reduced. A progressive disappearance of the Pt⁰-CO band was nevertheless observed, while the amount of CO₂ in the gas phase increased significantly. After 1.5 min of microwave irradiation (Fig. 4 C and D), the interaction of CO with platinum atoms was no longer observed, suggesting that all carbon monoxide molecules reaching the catalyst surface were immediately converted. This result shows that the catalyst was effectively heated under microwave irradiation to a temperature high enough to convert CO into CO₂. The absence of reaction in the absence of metal was checked by microwave heating of a zeolite sample with no metallic

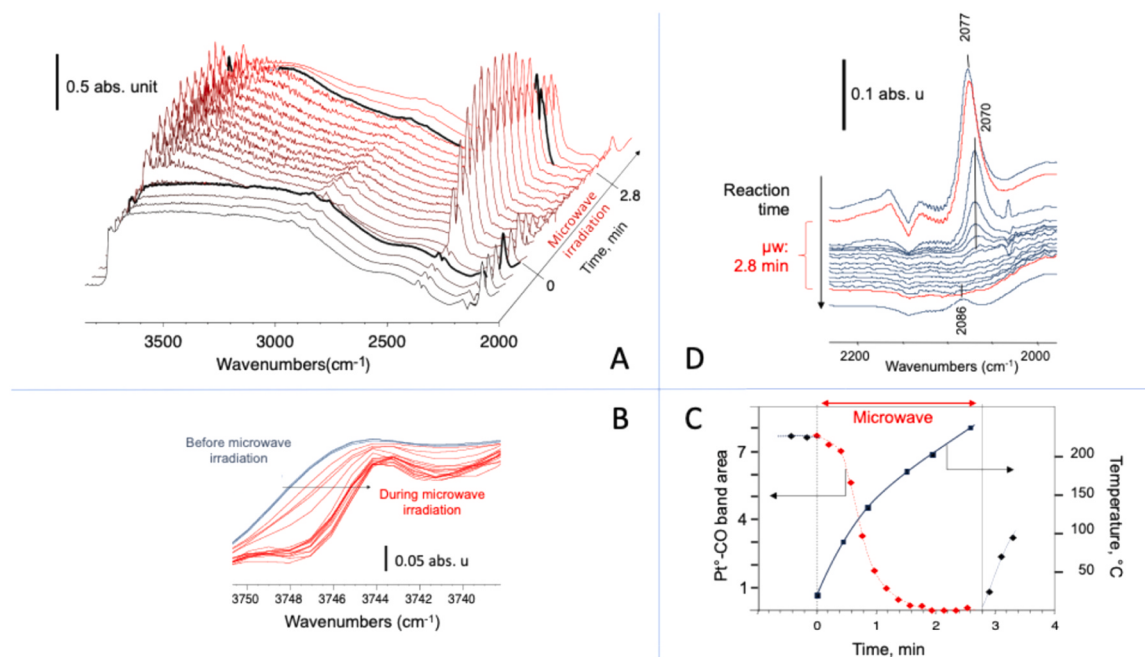


Fig. 4. A: IR spectra of the catalyst taken before, during and immediately after microwave irradiation. B: spectra of the catalyst in the silanol vibration region, prior to and during microwave irradiation (the arrow indicates the order in which the spectra were taken). C: area of the band due to CO-Pt interaction before, during and immediately after microwave irradiation, together with the estimated temperature of the solid. D: Position of the Pt⁰-CO band before, during and immediately after microwave irradiation (the arrow indicates the order in which the spectra were taken).

function. When discontinuing microwave irradiation, the signal-to-noise ratio was immediately recovered and the Pt⁰-CO band was restored. The area of the Pt⁰-CO band during the experiment is shown in Fig. 4C.

Given what was observed under conventional heating, it is concluded that the catalyst was heated to at least 150°C. The actual sample temperature was determined using the red-shift of the silanol band at 3745 cm⁻¹ during microwave irradiation. As mentioned earlier, the quality of the spectra decreased during microwave irradiation, particularly in the 3500 and 1650 cm⁻¹ regions because of microwave absorption by water molecules inside the cell. For this reason, the hydroxyl vibration region was not as well defined as under conventional heating. Nevertheless, the red-shift of the silanol band can still be observed (Fig. 4B). After baseline correction, a clear shift of the ν(OH) band was observed from 3746 to ca. 3744 cm⁻¹, with a maximum value for this red-shift of 1.9 cm⁻¹. According to the calibration obtained under conventional heating (Fig. 2), the maximum temperature variation was 217°C, which means the catalyst was heated from room temperature up to 230 – 240°C (Fig. 4C).

3.4. Indication for an electronic perturbation of Pt under microwave irradiation

The IR features for CO on Pt led to interesting new information on the behaviour of the metal under microwave irradiation, indicating a possible electronic effect on the metal reactivity unique to microwave. During microwave irradiation, the intensity of the ν(CO) vibration band for CO on Pt⁰ progressively decreased down to complete disappearance, showing the efficiency of the oxidation reaction and the consumption of surface CO under heating. The intensity of the ν(CO) absorption band for CO on Pt is commonly known to vary with temperature since it also affects CO adsorption equilibrium on Pt [23].

When the microwave was stopped and the temperature decreased back to its initial value, CO came back to the surface since it was continuously fed to the reactor and the ν(CO) band reappeared (Fig. 4D, bottom spectrum).

The frequency of the ν(CO) vibration band for CO on Pt⁰ atoms was

interestingly affected by the microwave irradiation. As seen in Fig. 4D, the band shifted to lower wavenumbers (from 2077 to 2070 cm⁻¹) during microwave irradiation and came back to higher wavenumbers (2086 cm⁻¹) after the microwave had stopped.

3.4.1. Mechanistic basis for CO band shift

IR monitoring of CO adsorption on supported metals is commonly used to measure metal particle size and dispersion [23]. This is simply done by measuring the maximum intensity of the ν(CO) vibration band when progressively introducing the gas on the reduced sample. A simple quantitative relationship between the band intensity and the amount of metallic adsorption sites per gram of catalyst is used to determine the number of accessible metal atoms on the solid and thus the particle size and dispersion. This was used in the present study to determine the 1.5 nm average particle size in the catalyst before the reaction.

Similarly to what is observed for other adsorbed species, the frequency of the ν(CO) vibration band is strongly affected by electron density on the CO adsorption site. There is however something peculiar, but well known, for CO on metals [16]. CO binds to the metal by donating electrons. At the same time, a retro-donation takes place from the metal d-orbitals to the CO molecule, populating an antibonding molecular orbital. A high electron density on the metal therefore induces a weakening of the CO bond and thus a lowering of the ν(CO) vibration frequency. Because of the relative amount of metal atoms in planes, edges and corners in small particles, with different electron densities on the metal atom, the average electron density on metal particles depends on the particle size: a link is known between the frequency of the ν(CO) vibration band and the particle size [20]. Coverage also influences the ν(CO) frequency since it slightly modifies electron density on the metal particle.

Several factors could therefore explain the various frequency shifts of the ν(CO) on Pt⁰ observed in the present work under (or after) MW irradiation:

- Shift to higher frequency after microwave irradiation. Sintering of the metal particles (because of strong heating, for example) would lead to a shift of the ν(CO) band to higher wavenumbers [27]. This

corresponds to what is observed here when MW has stopped: the very small shift of the $\nu\text{CO-Pt}^0$ band from 2077 cm^{-1} to 2086 cm^{-1} could indicate some moderate sintering and the appearing of Pt particles 2–5 nm large [27] (the intensity of the band grows back progressively after the microwave has stopped, as seen in Fig. 4C and on the bottom spectrum in Fig. 4D). Heating by microwave can be very strong in the neighbourhood of metal particles because of possible hotspots. Some moderate sintering is therefore expected here although the temperature of the zeolite support stays moderate. This particle size increase from 1.5 to 2–5 nm, detected here with IR features, is not surprising and this preliminary finding will need to be confirmed with TEM or by peak broadening in XRD.

- Shift to lower frequency during microwave irradiation. Decreasing CO coverage on the metal particle could progressively shift the $\nu(\text{CO})$ to lower wavenumbers. This would be expected here because of progressive oxidation of CO to CO_2 and CO disappearance on the surface, with a progressive shift of the band, linear with coverage. However, this is not what is observed here and the band changes immediately from one position to the other as soon as the sample is under microwave irradiation. This sudden band shift is therefore not due to progressive decrease of coverage, but is rather an indication that microwave induce a sudden increase of electron density on the external surface of the Pt particles. It would be unique evidence for a specific microwave effect in heterogeneous catalysis. This must be confirmed by other techniques and is a very interesting hint for further studies.

4. Conclusion

A new spectroscopic cell was designed for operando IR observation of heterogeneous catalysis under microwave irradiation. It was validated by comparing the oxidation of CO on Pt-H-BEA zeolite under conventional and microwave heating under the same flow conditions, in the same reactor. The temperature for complete oxidation of CO (150°C) was used as a benchmark to assess the efficiency of microwave heating. A useful temperature scale was provided by monitoring the frequency of the infrared band for the $\nu(\text{SiO-H})$ vibration of terminal silanols on the zeolite around 3745 cm^{-1} . This band shifts to lower wavenumbers linearly with the temperature increase. A final temperature of $230\text{--}240^\circ\text{C}$ was obtained under microwave irradiation.

During the course of the experiment, some moderate sintering of the platinum particles after microwave irradiation was observed (by the blue shift of the $\nu(\text{CO})$ on Pt^0), which indicates probable hotspots on the metal. At the same time, a surprising red-shift of the same band under irradiation might indicate a strong perturbation of electron density in the Pt particles by microwave.

CRediT authorship contribution statement

Frederic Thibault-Starzyk: Writing – original draft, Supervision, Resources, Project administration, Methodology, Investigation, Data curation, Conceptualization. **Ana Neto:** Writing – review & editing, Investigation, Data curation. **Sebastien Thomas:** Supervision, Methodology, Investigation, Conceptualization. **Gary Bond:** Writing – review & editing, Supervision, Methodology, Investigation, Conceptualization. **Carlos Henriques:** Writing – review & editing, Supervision, Methodology, Investigation, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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