

# Microstructural evolution and sintering of Pt nanoclusters on diatomite under CO oxidation conditions: An *operando* and 3D TEM study

Othmane Darouich<sup>1,2</sup>, Dris Ihiawakrim<sup>1</sup>, Sana Labidi<sup>3</sup>, Ovidiu Ersen<sup>1</sup>, and Walid Baaziz<sup>1</sup>  

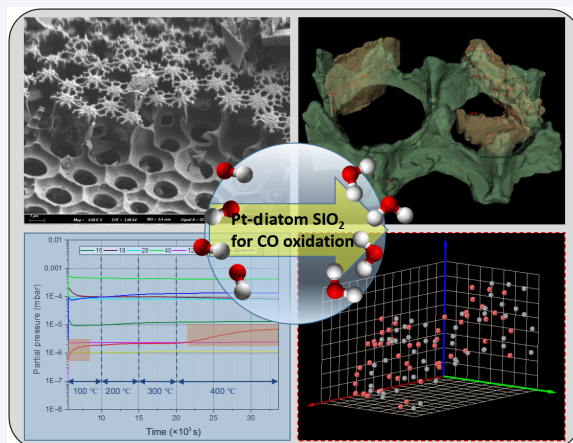
<sup>1</sup>Institute of Physics and Chemistry of Materials of Strasbourg (IPCMS), UMR 7504 CNRS-University of Strasbourg, 23 rue du Loess BP 43, 67034 Strasbourg cedex 2, France

<sup>2</sup>Laboratory of Chemistry of Condensed Matter of Paris (LCMCP), Sorbonne University-CNRS, 4 Place Jussieu, 75005 Paris, France

<sup>3</sup>Institute of Chemistry and Processes for Energy, Environment and Health (ICPEES), ECPM, UMR 7515 CNRS-University of Strasbourg, 25 rue Becquerel, 67087 Strasbourg Cedex 02, France

 Cite this article: Nano Research, 2026, 19, 94908808. <https://doi.org/10.26599/NR.2026.94908808>

**ABSTRACT:** In this work, we investigated the microstructural evolution and thermal stability of platinum (Pt) nanoparticles supported on natural diatomite bio-silica ( $\text{SiO}_2$ ) during catalytic carbon monoxide (CO) oxidation using advanced transmission electron microscopy (TEM) techniques. Pt nanoparticles with an average size of  $\sim 1$  nm were obtained by impregnating purified diatomite from *Coscinodiscus* sp. with a  $\text{Pt}(\text{acac})_2$  precursor and are preferentially located on the cribrum/foramen regions, where a native Fe/Al-rich oxide layer promotes selective anchoring. *Operando* TEM combined with mass spectrometry reveals that the Pt-diatomite catalyst is active from room temperature up to 400 °C, exhibiting fast reaction kinetics between 25 and 100 °C and at 400 °C. Three-dimensional electron tomography performed on the same Pt-diatomite grain before and after CO oxidation enables direct comparison of Pt nanoparticle positions and silica porosity, confirming the high structural stability of the catalyst under these conditions. The microstructural behavior was further examined up to 1000 °C under reactive atmosphere: The catalyst remains stable up to 600 °C, while at higher temperatures (600–800 °C) Pt nanoparticle sintering occurs as the Fe/Al-oxide layer degrades, leading to enhanced CO conversion up to  $\sim 950$  °C followed by partial deactivation at higher temperatures due to extensive sintering. These results provide direct nanoscale evidence of how a natural Fe/Al-rich interfacial layer governs the stability, sintering pathways and performance of bio-silica-supported Pt catalysts under severe CO oxidation conditions, outlining a general mechanism for the thermal stability and deactivation of bio-derived hierarchical silica supports in high-temperature oxidation catalysis.



**KEYWORDS:** CO oxidation, Pt nanoparticles, natural silica diatomite, *operando* transmission electron microscopy (TEM), electron tomography

## 1 Introduction

Carbon monoxide (CO) oxidation reaction has attracted significant attention due to its importance in both fundamental research and practical applications, especially in petrochemical processes, fuel

synthesis, gas masks, air purification, and the reduction of automotive emissions [1–6]. Among various catalysts for this reaction, platinum supported on silica ( $\text{Pt-SiO}_2$ ) exhibits prominent properties but also presents challenges [7–9]. The thermal stability of these catalysts is often limited due to the weak interaction between platinum and silica, which negatively affects platinum dispersion at elevated temperatures [10, 11]. The properties of silica significantly influence the behavior of supported platinum catalysts, as they vary widely in terms of surface area, pore and particle size, functional groups, and acid-base characteristics [9, 12–14]. The

Received: February 23, 2026; Revised: April 27, 2026

Accepted: May 4, 2026

 Address correspondence to [walid.baaziz@ipcms.unistra.fr](mailto:walid.baaziz@ipcms.unistra.fr)

dispersion and thermal stability of platinum are typically enhanced by incorporating metal oxide layers (e.g., Ti, Fe, and Al) onto the silica surface, which promotes the formation of Pt–O–M bonds [15–17]. The activity of Pt-based catalysts for CO oxidation is often limited at low temperatures due to the competitive adsorption of CO and O<sub>2</sub> on platinum active sites [18]. The strong binding of CO on the Pt surface reduces the availability of active sites for O<sub>2</sub> adsorption and activation, thereby hindering the overall reaction rate [19, 20]. To enhance the CO oxidation activity, oxygen supply from the support and/or promotion of platinum by iron oxide species have been shown to effectively facilitate O<sub>2</sub> activation [21–26]. Siani et al. reported that the temperature for total CO conversion over Fe-promoted PtFe/SiO<sub>2</sub> was reduced to 140 °C, compared to 230 °C for conventional Pt/SiO<sub>2</sub> prepared by incipient wetness impregnation [27].

Recently, natural diatomite bio-silica has attracted increasing research interest as a catalyst support for oxidation reactions due to its unique three-dimensional (3D) ordered and hierarchical porous structures, high specific surface area, excellent thermal stability, and chemical inertness [28–33]. Therefore, it presents an interesting alternative to synthetic silica materials. The frustule of such diatoms consists of amorphous silica containing a variety of inorganic residues usually involved in silicon biomineralization of live diatoms, especially oxides of Fe, Al, Ca, and Mg, which can serve as effective anchoring sites for nanoparticles (NPs) intended for catalytic applications [34–36]. In addition, diatomite usually requires purification treatments to remove impurities such as alkaline or alkaline earth carbonates and quartz. Only few studies have reported the use of such materials in catalysis. Zhang et al. employed palladium nanoparticles supported on diatomite to catalyze the Heck and Suzuki reactions [37]. Platinum nanoparticles supported on *Stephanopyxis turris* diatoms have been used as catalysts for redox reactions involving hexacyanoferrate and sodium thiosulfate [38]. Fisher et al. reported gold nanoparticles decorated on diatoms as effective catalysts for the oxidation of D-glucose [31]. For CO and hydrocarbon oxidation reactions, modified diatomite supported CuO–TiO<sub>2</sub> and Mn composites were reported by Savari et al. and Badii et al., respectively [39, 40].

In the current paper, we report the study of frustules of *Coscinodiscus* sp. diatom species [41] as catalytic supports for Pt NPs in the oxidation of CO. The improvement of their catalytic properties requires a deep characterization of their 3D nanometric nanostructure and subsequent behaviour in reaction conditions. The microstructural evolution of the resulting catalyst was obtained by advanced transmission electron microscopy (TEM) via the implementation of a quantitative approach that combines *operando* TEM with mass spectrometry and electron tomography (ET).

## 2 Experimental

### 2.1 *Coscinodiscus* diatomite specimen

The *Coscinodiscus* sp. diatomaceous frustules originate from a diatomite formation of the Sig Quarry, located in the Bas Chérif Basin in Algeria [42]. The diatomite was formed mainly of centrophycidae. These planktonic species lived in cold or cold temperate waters during the Messinian period [43]. The raw diatomite was found to contain traces of calcium carbonate (CaCO<sub>3</sub>), quartz, and iron oxide [44]. Purification treatment based on acid washing and sedimentation steps was then carried out to eliminate the CaCO<sub>3</sub> and quartz, respectively. 5 g of crude diatomite

was added to 300 mL of solution of 33% nitric acid and the mixture was stirred for about 17 h. The solution was then centrifuged and the diatomite was collected and washed to a neutral pH. 40 mL of distilled water was added to the solid deposit and the solution was left to decant for 13 min. The liquid part containing the structures of interest was collected and dried at 100 °C. More details are present in Ref. [42].

### 2.2 Pt-diatomite catalyst preparation

A freshly prepared Pt(acac)<sub>3</sub> solution in acetone was mixed with a dispersed diatomite in hexane. The Pt/diatomite ratio was adjusted to 2 wt.% which is the theoretical loading calculated for the synthesis (based on the precursor mass and targeted impregnation). Elemental analysis confirmed an actual Pt incorporation of 0.2 wt.% within the SiO<sub>2</sub> matrix of diatomite. The resulting mixture was then stabilized under air for 24 h at ambient temperature, then filtrated and dried under air at 40 °C for 17 h and finally calcined under air at 300 °C for 2 h.

### 2.3 Electron microscopy: TEM, scanning transmission electron microscopy (STEM), and energy-dispersive X-ray spectroscopy (EDS)

STEM analysis of Pt-diatomite catalyst was carried out using a JEOL 2100 FEG S/TEM microscope operated at 200 kV equipped with a probe spherical aberration corrector. The samples were dispersed by ultra-sonication in ethanol and deposited on a holey carbon coated TEM grids. The STEM images were carried out using a spot size of 0.13 nm, a current density of 140 pA, and a camera focal length of 8 cm, corresponding to inner and outer diameters of the annular detector of about 73 and 194 mrad, respectively. EDS maps and spectra of Si, Pt, Fe, and O were carried out with an EDS probe using a silicon drift detector (SDD) with a sensor size of 60 mm<sup>2</sup>.

### 2.4 *Operando* TEM and residual gas analyzer (RGA) mass spectrometry

Fragments of Pt-diatom sample were deposited onto an environmental cell (E-cell) from Protochips support, which was a thin ceramic heating membrane controlled with the “atmosphere” software in order to automatically adjust the temperature under different pressures and gas species. A second cell chip with a SiN membrane closed the thermal E-cell in the TEM holder, producing a thin gas cavity sealed with small o-rings maintaining the high vacuum in the TEM column. During the experiment, the sample was observed at a pressure of 1 atm (760 Torr) of argon to localize the zones that would be observed later, then the gaz (Ar, O<sub>2</sub>, and CO) was introduced and the sample was heated to the chosen temperatures. The exhaust line of the environmental TEM (E-TEM) holder was connected to an RGA.

For *operando* part, the sample was imaged in STEM mode with a probe size of 1 Å and a beam current of 35 pA. The electron dose rate was calculated as follows: the probe current of 35 pA, corresponding to  $2.18 \times 10^8$  electrons/s ( $I/q$ , where  $q = 1.602 \times 10^{-19}$  C). For typical area imaged with 1024 pixel  $\times$  1024 pixel image with a pixel size of 3.4 Å, the probed area was determined as  $A = N_{\text{pixels}} \times (\text{pixel\_size})^2 = 1048576 \times (3.4 \text{ Å})^2 = 1.21 \times 10^7 \text{ Å}^2$ . The resulting dose rate was  $J = (2.18 \times 10^8 \text{ e}^- \cdot \text{s}^{-1}) / (1.21 \times 10^7) = 18 \text{ e}^- \cdot \text{Å}^{-2} \cdot \text{s}^{-1}$ . Table S1 in the Electronic Supplementary Material (ESM) resumes the electron dose rates calculated for the different areas present in the figures.

## 2.5 Electron tomography

For the ET study, the E-cell containing the sample was introduced and fixed inside the specific sample holder destined to the ET experiments. The acquisition of tilt series was acquired using the tomography plug-in of the digital micrograph software, which controlled the specimen tilt step by step, the defocusing, and the specimen drift. The high angle annular dark field (HAADF) and bright field (BF) tilt series in the STEM were acquired by using the ADF and BF detectors and tilting the specimen in the angular range of  $\pm 51.5^\circ$  using an increment of  $1.5^\circ$  in the equal mode, giving thus a total number of images equal to 69 images in each series. The inner radius of the ADF detector was about 40 mrad, a relatively large value that allowed us to consider that the intensity in the corresponding images is proportional to the mean atomic number of the specimen in first approximation.

The recorded images of the tilt series were spatially aligned by cross correlating consecutive images using IMOD software. For the volume reconstruction, the algebraic reconstruction technique (ART) implemented in the TomoJ plugin working in the ImageJ software was used. Finally, the visualization and the analysis of the final volumes were carried out using the displaying capabilities and the isosurface rendering method available in the Slicer software.

## 3 Results and discussion

### 3.1 Microstructural properties of the Pt-SiO<sub>2</sub> diatom catalyst

#### 3.1.1 Size and crystalline structure of Pt NPs

The *Coscinodiscus* sp. diatomite analysed in this study corresponds

to a fraction of the purified natural bio-silica reported previously, which exhibits a three-dimensional hierarchical porosity [39]. The typical structural lattice consists of a central areola layer that supports a series of foramen layers displaying a hexagonal arrangement, as well as the simultaneous presence of cribrum layers. Nevertheless, it was observed that some regions of the pristine diatomite, particularly the cribrum layers, are often partially dissolved by the acidic treatment used to remove residual organic matter or to break due to their fragile nature [45]. SEM and TEM/STEM micrographs of the pristine *Coscinodiscus* sp. diatom, i.e., before platinum deposition, are shown in Fig. S1 in the ESM and reveal the presence of areola, cribrum, and foramen parts. The global architecture of the diatom is preserved after the synthesis of platinum nanoparticles on its surface. Nevertheless, in some cases the cribrum and foramen layers appear to be overlapped, although their positions remain fixed relative to the areola walls.

Figure 1 shows STEM micrographs recorded at different magnifications for the as-synthesized Pt-diatomite nanostructure (see also Fig. S2 in the ESM). Two populations of platinum NPs are observed, mainly on the surfaces of the cribrum and/or foramen layers. The most important amount of platinum is present in clusters and small atom clusters with sizes of a few ångströms (Fig. 1(d)), while some additional NPs with sizes of 3–4 nm are also detected. The average particle size, determined from more than 800 NPs, is  $1 \pm 0.9$  nm. The crystalline structure of platinum is examined by high-resolution STEM (HR-STEM) (Figs. 1(d)–1(f)), which clearly reveals the presence of well-crystallized particles. The measured interplanar distances of 0.23 and 0.19 nm correspond to the expected distances of the (111) and (200) planes of metallic platinum with a face-centred cubic (fcc) structure (JCPDS No. 03-065-2868).

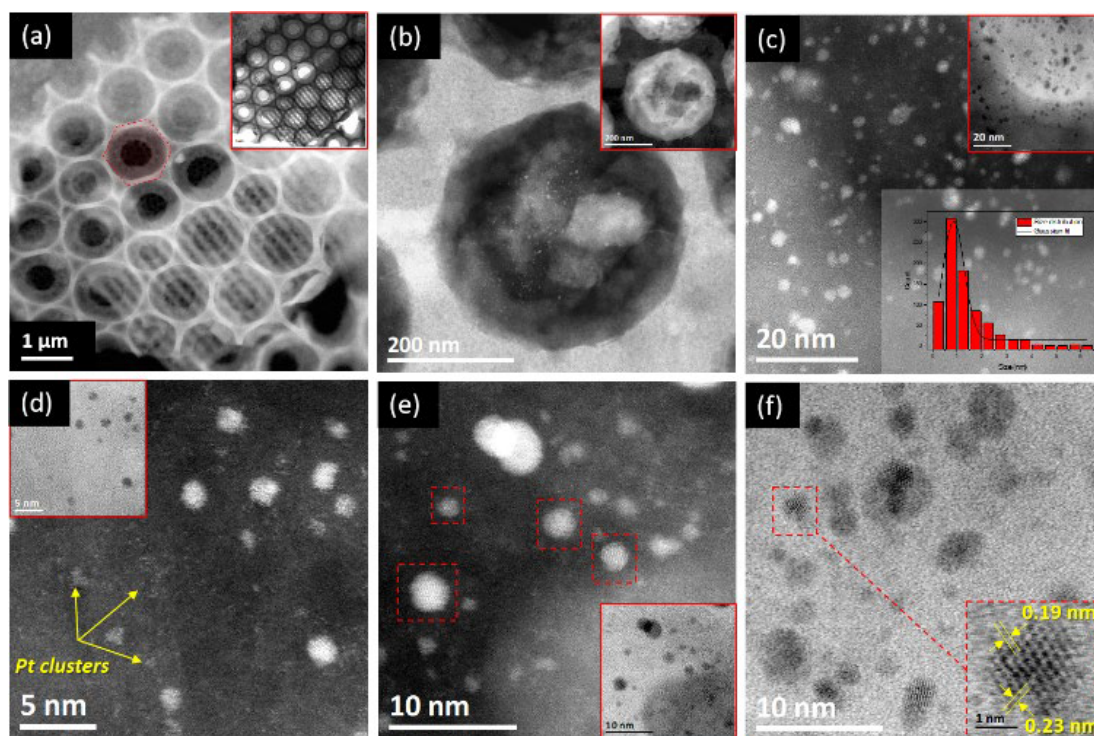


**Figure 1** ((a)–(d)) STEM ADF (with insets showing the corresponding STEM BF) micrographs of typical areas of the Pt-diatomite catalyst with different magnifications, showing the conservation of the *Coscinodiscus* sp. diatom after Pt impregnation and the presence of well dispersed Pt on its surface. In inset in (c), the size distribution of the Pt NPs fitted with a Gaussian function. (e) HR-STEM micrograph showing the crystalline structure of Pt NPs. (f) HR-STEM BF micrograph, with inset showing a zoom of the chosen area illustrating the interplanar distances on one typical Pt particle.

### 3.1.2 Preferential localization of Pt NPs on the cribrum and foramen layers

STEM images recorded on several catalyst grains reveal that most Pt nanoparticles anchor preferentially on the cribrum and/or foramen regions rather than the areola walls of the *Coscinodiscus* sp. diatom (Fig. S2 in the ESM). To precisely investigate this localization of platinum, electron tomography was performed on a representative grain of the Pt-diatomite catalyst.

Classical two-dimensional (2D) STEM images of the catalyst, which are projections of the real 3D volume, do not allow to unambiguously solve the localization of Pt nanoparticles on the different diatomite components. In contrast, an electron tomography analysis (Fig. 2) clearly shows that platinum selectively localizes on the cribrum and/or foramen regions (highlighted in yellow in the 3D model), with only a few nanoparticles on the areola walls (see also the 3D model animation in the ESM). The 3D analysis also reveals the internal porosity within the areolae walls, which consists of a microporous framework with pore sizes of 5–20 nm (Fig. 2(c)). To our knowledge, no prior studies have reported such preferential deposition of metal or metal oxide nanoparticles on specific diatom components. To understand the origin of this selective Pt NP deposition, chemical and structural analyses were performed using EDS and HR-STEM on representative regions of the diatomite components.

Figure 3(a) presents a comparative EDS study of cribrum/foramen and areola fragments. The results clearly reveal the presence of Fe, Al, and Mg on the cribrum/foramen regions, whereas the areolae consist solely of Si and O, with minimal amount of Fe consistent with pure silica. In a previous study, analysis of this diatomite identified amorphous silica, residual crystalline quartz, calcium carbonate (removed by acid treatment), and traces of Mg, K, Cl, and Fe [42]. TEM-EDS analysis in this study confirms these findings and precisely localizes residual Fe, Mg, and Al to the cribrum/foramen components (area 1 in Fig. 3(a)). Their contents vary across different regions of the cribrum/foramen layers. No Cl or K was detected in these areas.

Diatom cells are known to incorporate residual microcrystalline materials, such as oxides of Ca, K, P, Fe, Al, and Mg, from their environment or biological metabolism [46–49]; however, prior reports did not specify their precise locations. To our knowledge, this is the first report of the preferential localization of Fe, Al, and Mg oxides localized exclusively to the central cribrum/foramen region of *Coscinodiscus* sp. diatoms and, in return, their absence on the areola walls (area 2 in Fig. 3(a)).

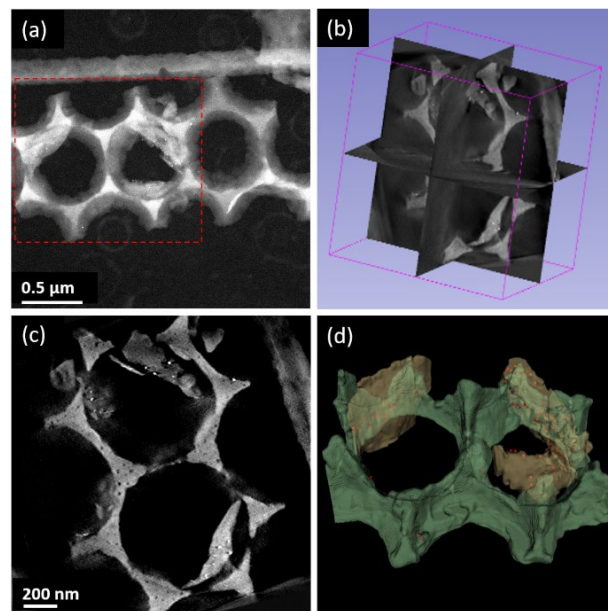


**Figure 2** Tomographic analysis realized on a representative grain of Pt-diatomite catalyst: (a) STEM ADF micrograph of the considered grain (the area considered for the 3D analysis is highlighted by a red square). (b) 3D insight through the calculated volume. (c) Typical (XY) slice extracted from the calculated volume, showing the presence of the Pt NPs in the overlapped cribrum/foramen layers and their absence on the areola walls. (d) 3D model of the Pt-diatomite grain present in (a), with Pt NPs represented in red.

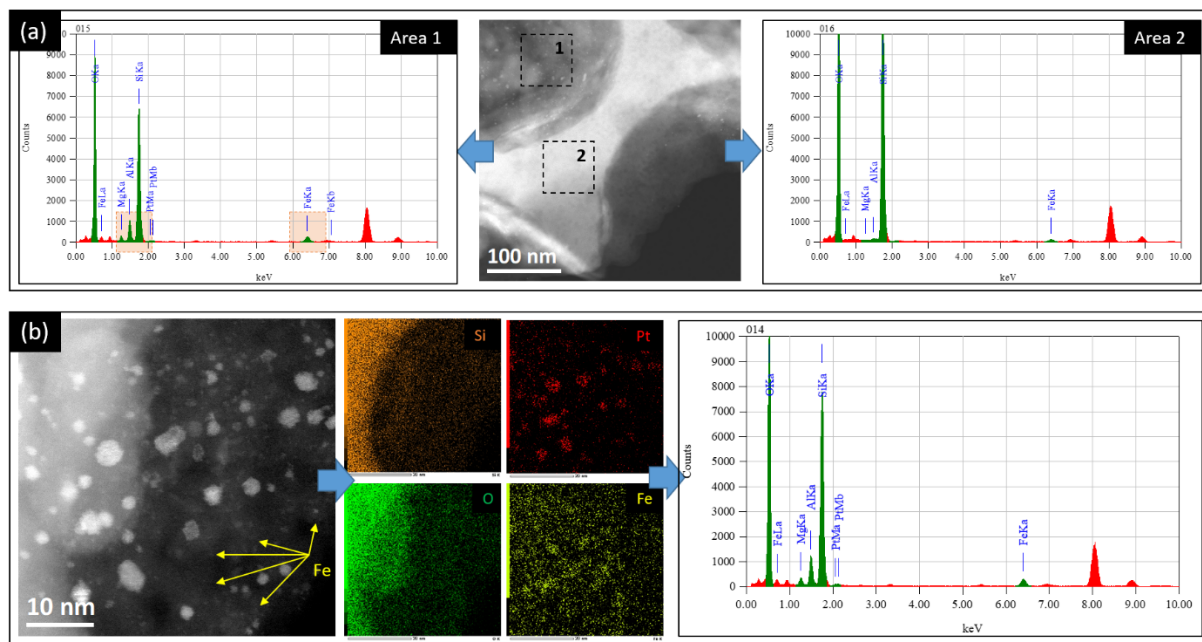


**Figure 3** (a) Typical EDS spectra acquired on cribrum/foramen area (area 1) and areola wall (area 2). (b) From left to right: representative area of the cribrum/foramen rich in Pt NPs and the corresponding elemental maps of Si, Pt, O and Fe, together with the associated global EDS spectrum.

Among the oxides identified previously in the literature and in this work by EDS, iron oxide is particularly noteworthy, as it may explain the preferential Pt adsorption on the cribrum/foramen layers. Silica surfaces, being neutral, interact weakly with metal nanoparticles and typically require functionalization to serve as catalytic supports. Iron oxide is commonly used to modify silica surfaces prior to Pt deposition and plays a key role in the adsorption, stabilization, and activity of noble metals in CO oxidation reactions. Other identified oxides on areola walls, mainly Al and Mg, could display affinity to Pt nanoparticles, since their presence enhances surface basicity. It was reported that partially SiO<sub>2</sub> overcoated Al<sub>2</sub>O<sub>3</sub> developed tunable affinity to chloroplatinic complex: Accessible Al sites display strong electrostatic adsorption for Pt [24, 45, 46].

EDS analysis shows that Al, Mg, Si, and O are evenly distributed across the analysed area, whereas Fe forms denser entities on which Pt nanoparticles selectively anchor (white contrast indicated by yellow arrows in Fig. 3(b)).

To confirm the composition of these entities as iron oxide phases and determine their crystalline structure, representative areas were characterized by HR-STEM imaging and analysed by fast Fourier transform (FFT). Figures 4 shows HR-STEM and FFT micrographs of a typical cribrum/foramen layer region identified as Fe-rich by EDS mapping; notably, EDS allows to detect Fe and Si as major elements in this local area (Fig. 4(a)). The FFT inset (Fig. 4(b)) reveals a crystalline structure with multiple domains of distinct crystallographic orientations, where measured interplanar distances of 0.44 and 0.27 nm can be better assigned to those of  $\beta$ -Fe<sub>2</sub>O<sub>3</sub> (JCPDS No. 01-076-1821) than to their crystalline SiO<sub>2</sub> counterparts (JCPDS No. 00-040-1498). This is consistent with the EDS results and with the previous literature [40, 50–52]. In contrast, similar analysis of areola surfaces confirms their amorphous character (Figs. 4(c) and 4(d)), as expected for the dominant silica component of diatomite.

Based on these analyses, the selective deposition of platinum

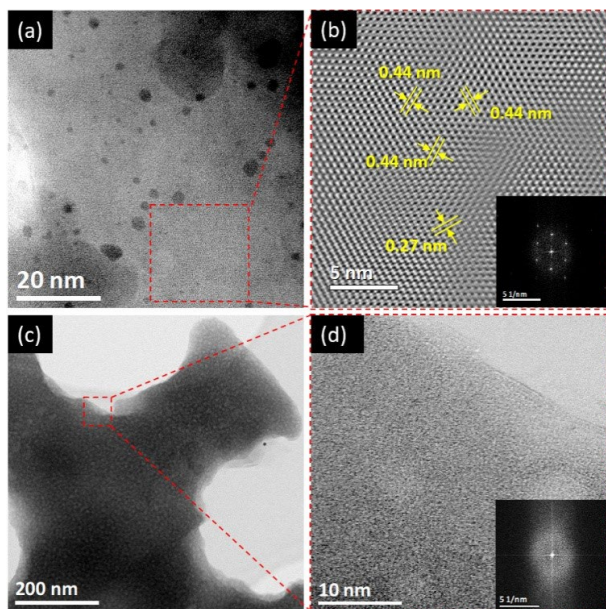


**Figure 4** ((a)–(d)) HR-STEM images showing ((a) and (b)) the crystalline structure of cribrum/foramen layer and ((c) and (d)) the amorphous character of the areola walls. Insets in ((b) and (d)) showing the correspondent fast Fourier transform micrographs.

nanoparticles on the cribrum/foramen components rather than the areola walls of diatomite arises from key factors. First, residual metal oxides, particularly iron oxide, on the cribrum/foramen surfaces alter the silica state, providing a natural surface modification. Literature shows that noble metal/oxide synthesis on silica, which initially bears only silanol groups (Si–OH), requires surface modification with oxides like iron oxide to create an active interface that enhances fixation, dispersion, and stability of metals such as Pt or Au [24, 50, 53]. Prior studies typically deposit metal/oxide nanoparticles on acid-treated or oxide-coated diatomite (e.g., with TiO<sub>2</sub> or ZrO<sub>2</sub>) or iron oxide to promote adsorption [37, 54]. Additionally, platinum deposition is influenced by porosity: It occurs at macropore interfaces contacting the cribrum/foramen, regions exposed to the cell interior during the diatom's life, rather than within the microporous interiors of the areolae.

### 3.2 Pt-diatomite evolution during CO oxidation reaction: *Operando* TEM and electron tomography

The catalytic behavior of Pt-diatomite and its microstructural evolution during CO oxidation were investigated under atmospheric pressure using *operando* TEM coupled with *in-situ* RGA analyses. The E-TEM cell, loaded with the catalyst sample, was initially purged at 100 Torr of argon to remove residual water and/or solvent traces. To mimic CO oxidation conditions, the catalyst was calcined in air at 300 °C for 3 h, then cooled to 25 °C; a reactant mixture (10% CO, 20% O<sub>2</sub>, and 70% Ar) was introduced at 0.1 sccm, heated to 400 °C at 5 °C·min<sup>−1</sup>, with 1 h held at 100, 200, and 300 °C.

The RGA, connected at the E-TEM cell outlet, enabled *in-situ* mass spectrometric analysis of the product gas, thereby tracking catalytic activity variations during the reaction. Monitored species included CO, O<sub>2</sub>, CO<sub>2</sub>, Ar, H<sub>2</sub>O, and N<sub>2</sub> (air); their *m/z* values are listed in Table S2 in the ESM. Figure 5 illustrates the *in-situ* evolution of their partial pressures as a function of time/temperature during CO oxidation over Pt-diatomite. Upon heating the sample from ambient temperature, O<sub>2</sub> fragment signals (corresponding to *m/z* ratios of 16 and 32) decreased rapidly, while the CO<sub>2</sub> signal (*m/z* = 44) increased sharply (the first highlighted region in Fig. 5). These changes indicate high CO oxidation activity from room temperature to 100 °C. The CO signal (*m/z* = 28) is challenging to interpret due to its overlap as both reactant and CO<sub>2</sub>

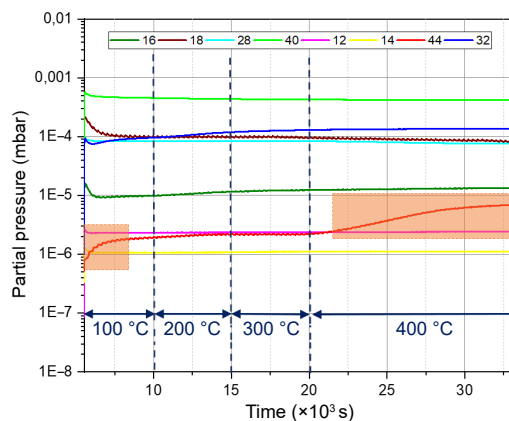


**Figure 5** Time evolution of partial pressure of the molecules present in the reaction medium as a function of time acquired by RGA during the *in-situ* reaction of CO oxidation over the Pt-diatom catalyst.

fragment. Catalytic activity increased until stabilizing after 1 h at 200 °C. CO<sub>2</sub> partial pressure remained stable between 200 and 300 °C, then increased again upon reaching 400 °C with slower kinetics than from ambient temperature (the second highlighted region in Fig. 5).

While environmental TEM cells enable *operando* monitoring of microstructural evolution under reactive atmospheres, they are not optimized for quantitative standard catalytic metrics measurements like turnover frequency (TOF) or precise conversion rates with the same rigor as in a dedicated catalytic reactor [25]. Given the preparation method (deposition of a few microliters of sample suspension onto a few-micron cell area) and the very low cell-controlled gas flow (~1 sccm, geometry- and catalyzer place-dependent), the reactive sample mass can be only roughly estimated, of the order of microgram. Thus, this setup primarily provides qualitative insights into structure–performance relationships. The microstructural evolution of Pt-diatomite during calcination and CO oxidation was investigated *in-situ* by imaging multiple catalyst areas. Figure 6 presents STEM images of two representative areas at varying magnifications: at 25 °C under vacuum, after a calcination (300 °C in air, 3 h), and after 1 h of CO oxidation at 100 and 400 °C. Additional areas are shown in Fig. S3 in the ESM.

STEM images reveal that Pt-diatomite remained highly stable during calcination and CO oxidation up to 400 °C, with no Pt sintering or coalescence observed. Pt NP size and positions, including those of small clusters, showed no variation except in isolated cases (yellow square, Fig. 6), likely due to electron beam effects prominent at high magnification. The electron beam effect was evaluated by imaging several areas only at 25 °C under vacuum and at the end of reaction (not during the thermal treatment), and similar behaviour of Pt NPs on other diatom areas was observed. It should be noted that the electron beam was kept off during the *operando* experiment and was only switched on for image acquisition once the target temperature was reached. The dose rates calculated for the followed areas in this work are illustrated in Table S1 in the ESM and range from 2 to 436 e<sup>−</sup>·Å<sup>−2</sup>·s<sup>−1</sup>. Lunkenbein et al. showed that unsupported platinum nanoparticles remained stable

for 1 h under continuous irradiation at a dose rate of 450 e<sup>−</sup>·Å<sup>−2</sup>·s<sup>−1</sup> under *operando* conditions similar to ours (a CO/O<sub>2</sub>/He mixture up to 400 °C at atmospheric pressure inside an *operando* TEM cell) [55].

To quantitatively assess Pt NP stability during CO oxidation, electron tomography series were acquired on the same Pt-diatom grain before and after thermal treatment. NP size distributions and inter-NP distances were extracted from the resulting 3D volumes for direct comparison. Figure S4 in the ESM shows representative STEM images and 2D slices from the reconstructed 3D volumes of the Pt-diatom grain before and after *in-situ* reaction. This approach provides quantitative 3D insights into morphological evolution, including Pt NP size, displacement, and silica pore dimensions. It is worthy to note that in this study, only Pt NPs larger than ~2 nm were analyzed, as smaller particles fell below the resolution limit of a classical tomographic analysis. Figures 7(a) and 7(b) show the 3D positions of Pt NPs in the investigated Pt-diatom grain before (white) and after (red) thermal treatment. The Pt NPs exhibit essentially the same locations in both reconstructed volumes, indicating their stability during the experiment.

From these 3D coordinates, the distances between each Pt NP and its neighbors were calculated, along with the distribution function  $N(r)$ , defined as the number of Pt NPs located at a distance  $r$  from a given particle. The pair distribution function  $g(r)$  was then obtained by dividing  $N(r)$  to the surface area of a spherical shell of radius  $r$ , that is  $4\pi r^2 dr$ . The resulting distributions (Fig. 7(c)) demonstrate that the interparticle distances between Pt NPs remain unchanged after thermal treatment, confirming the absence of significant nanoparticle migration and/or agglomeration. Importantly, identical reconstruction parameters, filtering procedures, and segmentation criteria were applied to both tomographic datasets to ensure a robust comparison with a reduced influence of some experimental conditions or image processing artefacts.

This approach also enables quantification of pore size distributions within areola walls (Fig. 7(d)), inaccessible through conventional STEM imaging (Fig. S4 in the ESM). We can observe that individual pore sizes exhibit minor variations, but the average

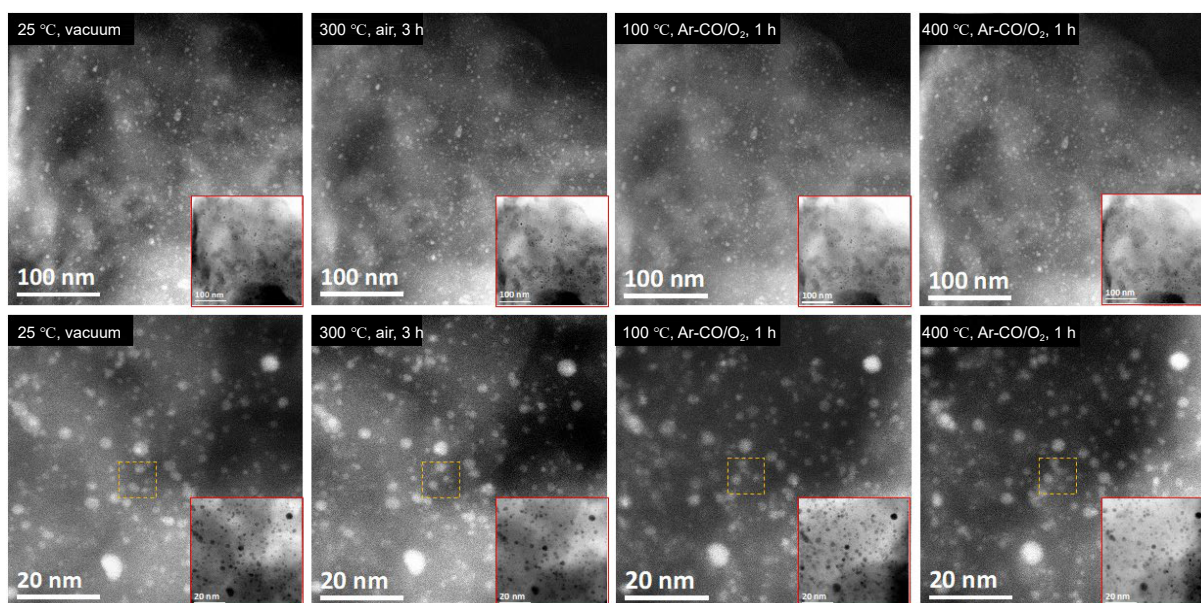


**Figure 6** STEM images of two typical areas of Pt-diatom catalyst followed *in-situ* during the calcination treatment and the CO oxidation reaction until 400 °C.

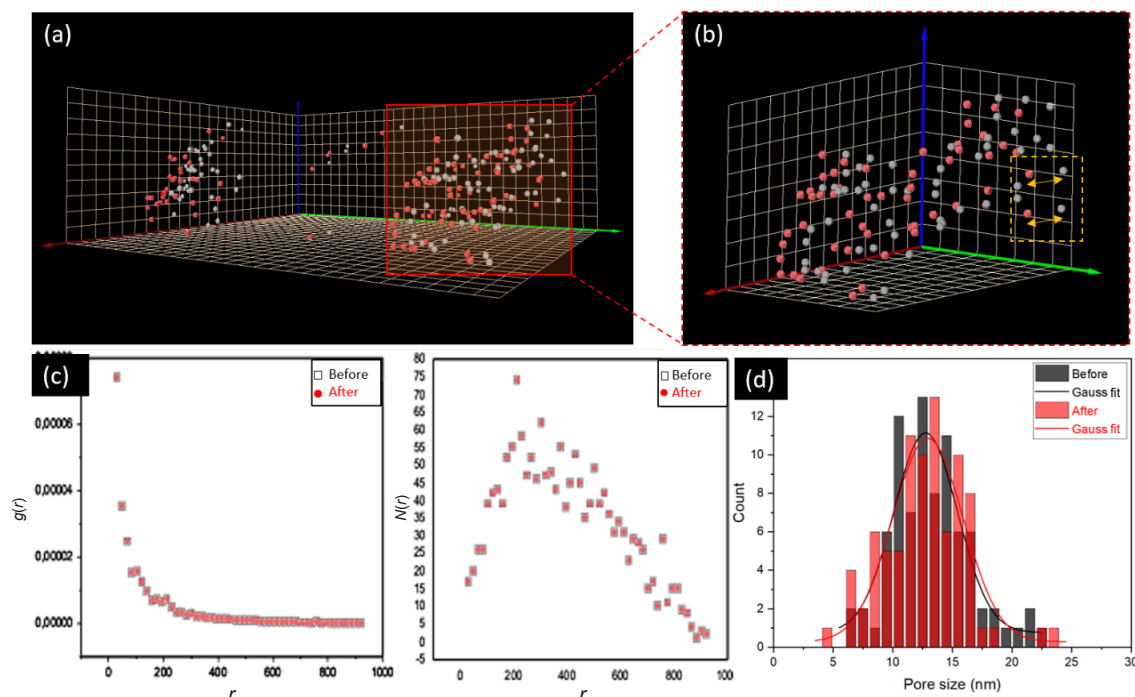


**Figure 7** (a) 3D position of the Pt NPs obtained from the tomographic data acquired on the same Pt-diatom grain (shown in Fig. 2) before and after the *in-situ* CO oxidation reaction. (b) Zoom on the highlighted area in (a), the distances shown by yellow arrows indicated the spatial shift between the two 3D volumes. (c) Comparison of the distributions of the distances between the neighbours Pt NPs before and after *in-situ* treatment. (d) Comparison between the areola pores size distributions before and after the *in-situ* treatment.

diameter remains unchanged after thermal treatment. In the literature, it was reported that diatom silica retains structural integrity and porosity up to 800–900 °C under dry oxidative conditions due to its amorphous structure and absence of low-melting phases; cristobalite crystallization occurs only above 1000 °C [56, 57].

### 3.3 Thermal stability of Pt-diatom during the catalytic oxidation of CO until 1000 °C by *operando* TEM coupled to RGA mass spectrometry

A second TEM *operando* experiment was performed to investigate the thermal stability of the Pt-diatom catalyst up to 1000 °C. This more fundamental study aims to have an insight on the microstructural evolution of the catalyst in “extreme” conditions of temperature under similar conditions of CO oxidation reaction. The temperature was ramped with an increment of 100 °C, with isothermally holding for 1.5 h at each step until CO<sub>2</sub> partial pressure stabilized. Figure 8 shows the stabilized CO<sub>2</sub> partial pressure as a function of temperature up to 1000 °C, along with its evolution over time between 500 and 1000 °C. The complete RGA spectra of CO<sub>2</sub>, CO, Ar, and O<sub>2</sub> partial pressures versus temperature and time, from ambient to 1000 °C, are presented in Fig. S5 in the ESM.

Up to 400 °C, the catalyst exhibits a behavior consistent with the previous experiment: A stable activity with rapid CO<sub>2</sub> partial pressure increases between 25 and 200 °C and from 400 °C onward. Note that measured partial pressures vary with catalyst mass in the E-cell, which differs across preparations. From 500 to 900 °C, CO<sub>2</sub> partial pressure rises with temperature, primarily reflecting thermal CO cracking rather than Pt NP catalytic activity, as confirmed by reference experiments using catalyst-free conditions [58]. A high reaction rate occurs between 600 and 800 °C. At 1000 °C, CO<sub>2</sub>

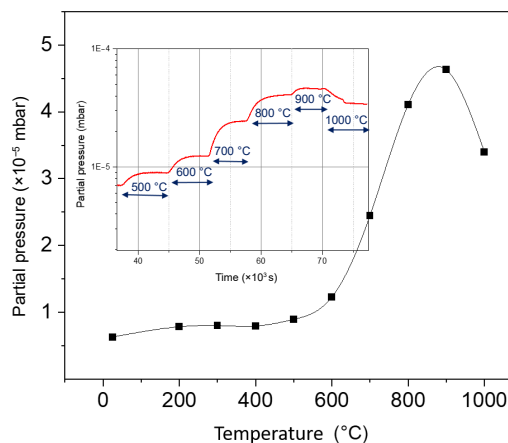


**Figure 8** Partial pressure acquired by RGA analysis of the CO<sub>2</sub> molecule ( $m/z = 44$ ) produced by the CO oxidation reaction as a function of temperature, and as a function of time (inset), revealing the kinetic of reaction.

partial pressure decreases and then stabilizes. In literature, it was reported that thermal cracking of CO ( $2\text{CO} \rightarrow \text{C} + \text{CO}_2$ ) becomes significant above 500–600 °C under oxidative conditions, with rates accelerating rapidly beyond 700 °C, i.e., at the range of 600–900 °C where thermal decomposition dominates over heterogeneous catalysis.

During thermal treatment, STEM images were acquired from several sample areas to characterize the evolution of Pt NPs and diatom silica. Figure 9 presents a representative area imaged at 25 °C under vacuum, after calcination at 300 °C in air, and during CO oxidation at 700 and 1000 °C. Additional areas are shown in Figs. S6 and S7 in the ESM.

The catalyst remains stable up to 600 °C. Above this temperature, high-resolution STEM imaging reveals damage to the iron oxide

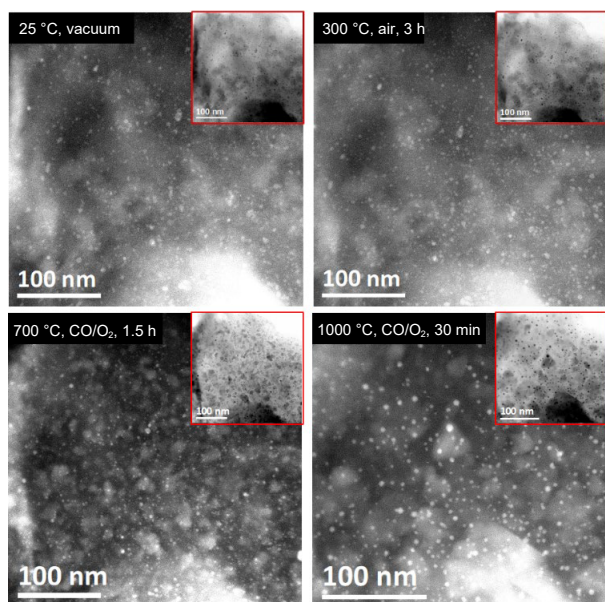


**Figure 9** STEM images of a typical area of Pt-diatom catalyst followed *in-situ* during a CO oxidation reaction until 1000 °C.

layer originally homogeneously distributed on the silica surface, as confirmed by prior EDS mapping. At 700 °C, iron oxide entities (10–20 nm) exhibit sintering, promoting Pt NP mobility and explaining the enhanced CO oxidation kinetics observed between 600 and 800 °C added to the thermal cracking. It should be noted that, in our *operando* TEM setup, the catalyst mass and gas residence time in the environmental cell are not known with sufficient accuracy to allow standard catalytic quantification. For the same reason, performing true blank or reference experiments under strictly identical conditions, to compare catalytic effects, is not really relevant. This explains why the contribution of possible thermal effects can only be discussed qualitatively here and would need to be addressed quantitatively in a dedicated reactor study. Loss of this anchoring  $\text{Fe}_2\text{O}_3$  layer likely induces sintering of the smallest Pt

clusters. High-resolution analysis of Pt NP size distributions (Fig. S7 in the ESM) shows stability up to 600 °C (average size of  $1.1 \pm 0.2$  nm), followed by increases to  $1.4 \pm 0.3$  nm at 800 °C and  $2.4 \pm 0.4$  nm at 900 °C. Despite sintering, most of NPs remain in the range of 2–3 nm below 900 °C. In this temperature range (500–800 °C), it is worthy to note that the observed “damage” of iron based layer could result from the  $\beta\text{-Fe}_2\text{O}_3$  to  $\alpha\text{-Fe}_2\text{O}_3$  transition, rather than pure phase decomposition [59]. Interactions with diatom  $\text{SiO}_2$  may locally lower these thresholds.

*In-situ* STEM video recordings (Videos S1 and S2 in the ESM) reveal Pt NPs sintering mechanisms at high temperature. Figure 10 shows STEM frames extracted from Video S1 in the ESM, capturing Pt NPs displacements on the cribrum/foramen surface at 1000 °C. Unfortunately, EDS analysis could not be performed at this temperature (and more generally above 450 °C) due to additional X-ray scattering in the gas phase and contamination from window degassing, preventing a reliable determination of the chemical species present during the *operando* monitoring. At this temperature, Pt NPs remain stable, preserving small particles (2–3 nm) without excessive sintering. The migration of the platinum was found to be directly related to the spatial distribution of the Fe-based entities, which are in movement on the  $\text{SiO}_2$  surface. In area 1 (Fig. 10), two Pt NPs destabilize upon contact with a migrating Fe entity (dark shadow), undergoing complete sintering after 88 s; the resulting particle then stabilizes without further Fe contact. In area 2, a Fe-rich zone similarly attracts and sinters Pt NPs between 41 and 88 s. In the Video S2 in the ESM, we can see that Pt was under thermal agitation but remains stable (not sintering) in the absence of iron-based entities. The observed mechanism of Pt sintering herein is consistent with particle migration and coalescence (PMC) rather than Ostwald ripening. In PMC, the NPs themselves migrate and merge upon contact, which matches with our observations of discrete Pt nanoparticles moving across the support when they are in the contact with Fe based entities and coalescing upon collision.

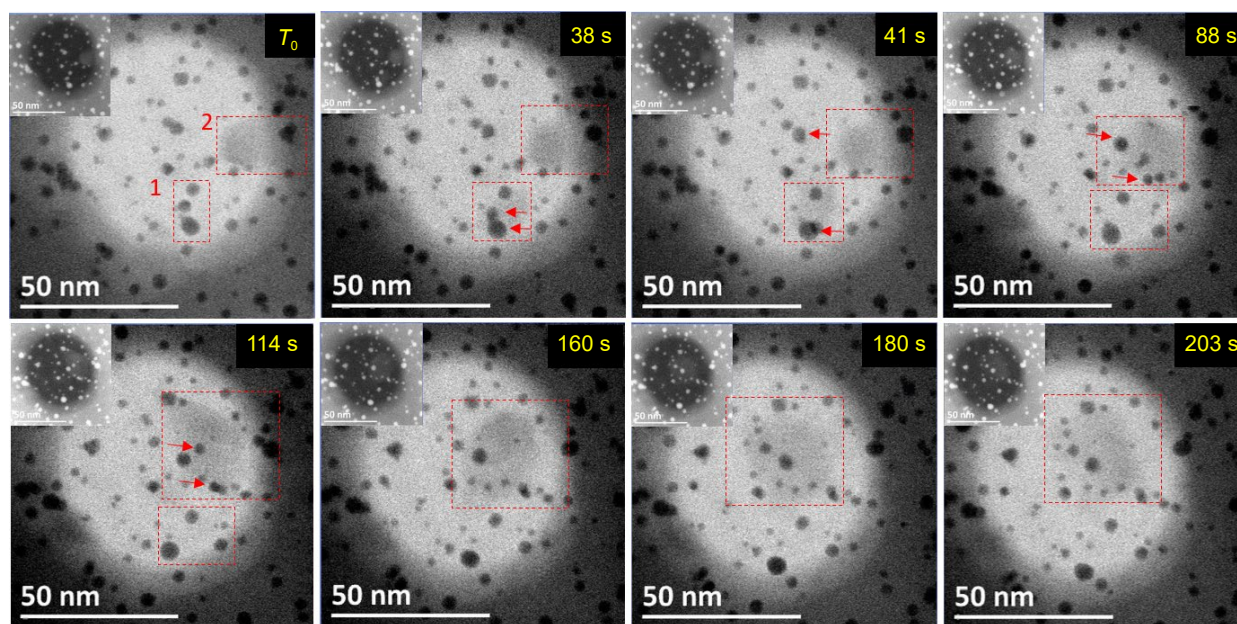


**Figure 10** STEM images of a typical area of Pt-diatom catalyst *in-situ* followed during the CO oxidation reaction at 1000 °C, showing the sintering mechanism of Pt NPs on the Fe-rich areas.

## 4 Conclusions

In this study, by using electron microscopy, we investigated the microstructure and the structural evolution of platinum nanoparticles synthesised on diatom silica via an impregnation method using  $\text{Pt}(\text{acac})_2$  as a precursor. Electron tomography, EDS spectroscopy, and high resolution imaging allowed to observe that platinum nanoparticles display an average size of 1–2 nm and are preferentially anchored on cribrum and/or foramen parts of diatom due to the presence of crystalline structure  $\beta\text{-Fe}_2\text{O}_3$  on the silica.

Quantitative analyses through *operando* TEM and electron tomography show that the synthesized Pt-diatom demonstrates exceptional thermal and microstructural stability up to 400 °C under oxidative conditions of CO oxidation reaction due to strong interaction of Pt with the iron oxide layer. The catalyst was found to be active starting from ambient temperature until 400 °C.

At higher temperatures, the preferential Pt NP deposition on Fe-rich diatom regions maintains optimal 1–2 nm of sizes and positions until 600 °C. Above 600 °C, the  $\text{Fe}_2\text{O}_3$  phase transitions ( $\beta \rightarrow \alpha$ ) and the sintering drives controlled Pt NP coalescence (to 2.4 nm at 900 °C), boosting kinetics alongside thermal CO cracking (dominant > 600 °C). The iron oxide transition was found to trigger Pt sintering at such high temperatures through a mechanism consistent with PMC. Diatom silica preserves hierarchical porosity (areola pores and cribrum/foramen) up to 900 °C, outperforming synthetic  $\text{SiO}_2$  supports. The diatom-derived architecture allowed thus a good NP confinement via dynamic  $\text{Fe}_2\text{O}_3$  anchoring, offering a bioinspired pathway for high-temperature catalyst design in exhaust treatment and syngas processing.

**Electronic Supplementary Material:** Supplementary material (electron dose rates, SEM and STEM images, mass/charge of gas molecules, partial pressure evolution, and size distributions of Pt nanoparticles) is available in the online version of this article at <https://doi.org/10.26599/NR.2026.94908808>.

## Data availability

All data needed to support the conclusions in the paper are presented in the manuscript and the Electronic Supplementary Material. Additional data related to this paper may be requested from the corresponding author upon request.

## Acknowledgements

The authors would like to thank Dr. Saliha Haddoum (École Nationale Polytechnique d'Alger, rue des Frères Oudak, 16200 El Harrach, Algeria) for kindly providing the diatomite sample used in this work. The authors also wish to thank Prof. Corinne Petit for her valuable help, insightful advice, and careful reading of the manuscript.

## Declaration of competing interest

All the contributing authors report no conflict of interests in this work.

## Author contribution statement

O. D.: TEM measurement, data curation. D. I.: Data curation,

experimental design. S. L.: Materials design/synthesis, elemental analysis. O. E.: Project administration, funding acquisition. W. B.: TEM measurement, data curation, project administration, writing manuscript. All the authors have approved the final manuscript.

## Use of AI statement

None.

## References

- [1] Ngorot Kembo, J. P.; Wang, J. Y.; Luo, N.; Gao, F. Y.; Yi, H. H.; Zhao, S. Z.; Zhou, Y. S.; Tang, X. L. A review of catalytic oxidation of carbon monoxide over different catalysts with an emphasis on hopcalite catalysts. *New J. Chem.* **2023**, *47*, 20222–20247.
- [2] Xie, S. H.; Lu, Y.; Ye, K. L.; Tan, W.; Cao, S. F.; Wang, C. Y.; Kim, D.; Zhang, X.; Loukusa, J.; Li, Y. B. et al. Enhancing the carbon monoxide oxidation performance through surface defect enrichment of ceria-based supports for platinum catalyst. *Environ. Sci. Technol.* **2024**, *58*, 12731–12741.
- [3] Teymoori, S. M.; Alavi, S. M.; Rezaei, M. Catalytic oxidation of CO over the  $\text{MO}_x\text{-Co}_3\text{O}_4$  (M: Fe, Mn, Cu, Ni, Cr, and Zn) mixed oxide nanocatalysts at low temperatures. *Sci. Rep.* **2025**, *15*, 25808.
- [4] Han, M. D.; Wang, Y. J.; She, X. F.; Zhang, Z.; Li, X. H.; Guo, Z. H. Recent progress in CO oxidation over non-precious-metal catalysts. *Ind. Eng. Chem. Res.* **2025**, *64*, 36–52.
- [5] Dey, S.; Mehta, N. S. Low temperature catalytic conversion of carbon monoxide by the application of novel perovskite catalysts. *Sci. One Health* **2022**, *1*, 100002.
- [6] Fang, Y. F.; Chen, X. H.; Liu, K. J.; He, Y. C.; Li, X. K.; Li, Y. N.; Yuan, Z. X.; Ye, J.; Liu, H.; Zhang, Y. B. et al. Facet-engineered Pt redispersion on  $\text{CeO}_2$ : Unraveling morphology and Pt density effects for tailored CO and  $\text{C}_3\text{H}_8$  oxidation catalysis. *ACS EST Eng.* **2026**, *6*, 510–523.
- [7] Garnier, A.; Sall, S.; Garin, F.; Chetcuti, M. J.; Petit, C. Site effects in the adsorption of carbon monoxide on real 1.8 nm Pt nanoparticles: An infrared investigation in time and temperature. *J. Mol. Catal. A: Chem.* **2013**, *373*, 127–134.
- [8] Demirdjian, B.; Ozerov, I.; Bedu, F.; Ranguis, A.; Henry, C. R. CO and  $\text{O}_2$  adsorption and CO oxidation on Pt nanoparticles by indirect nanoplasmonic sensing. *ACS Omega* **2021**, *6*, 13398–13405.
- [9] Taleblou, M.; Farnesi Camellone, M.; Fabris, S.; Piccinin, S. CO oxidation over platinum nanoclusters: Unraveling the role of the cluster size and the supporting surface. *J. Phys. Chem. C* **2023**, *127*, 21132–21149.
- [10] Park, J. H.; Cho, J. H.; Kang, S. E.; Cho, K. H.; Lee, T. W.; Han, H. S.; Shin, C. H. Low-temperature CO oxidation over water tolerant Pt catalyst supported on Al-modified  $\text{CeO}_2$ . *Korean J. Chem. Eng.* **2013**, *30*, 598–604.
- [11] Fang, Y. F.; Han, X. Y.; Liu, K. J.; Yuan, Z. X.; Li, Y. N.; Tao, S. Y.; Zhang, Z. S.; Rao, C.; Fu, Q.; Peng, G. et al. Current-assisted dual-atom catalyst sequentially boosts low-temperature propane combustion through atomic relay. *Nat. Chem.* **2026**, *18*, 445–456.
- [12] Park, S. K.; Kim, K. D.; Kim, H. T. Preparation of silica nanoparticles: Determination of the optimal synthesis conditions for small and uniform particles. *Colloids Surf. A: Physicochem. Eng. Aspects* **2002**, *197*, 7–17.
- [13] Chen, J. F.; Ding, H. M.; Wang, J. X.; Shao, L. Preparation and characterization of porous hollow silica nanoparticles for drug delivery application. *Biomaterials* **2004**, *25*, 723–727.
- [14] An, K.; Alayoglu, S.; Musselwhite, N.; Plamthottam, S.; Melaet, G.; Lindeman, A. E.; Somorjai, G. A. Enhanced CO oxidation rates at the

- interface of mesoporous oxides and Pt nanoparticles. *J. Am. Chem. Soc.* **2013**, *135*, 16689–16696.
- [15] Jeong, E. S.; Hwang, I. H.; Han, S. W. Dispersion and stability mechanism of Pt nanoparticles on transition-metal oxides. *Sci. Rep.* **2022**, *12*, 13652.
- [16] Ke, W.; Lee, I.; Zaera, F. Metal-oxide interface sites created using atomic layer deposition and tested for CO oxidation. *ACS Catal.* **2025**, *15*, 12281–12292.
- [17] He, Y. M.; Chen, J.; Xie, Y. J.; Hu, Q. S.; Wang, L. J.; Liu, Y.; Xu, X. L.; Xu, B. W.; Zeng, F. Effects of Mg on CO<sub>2</sub> hydrogenation on PtCoAl catalysts. *Catalysts* **2025**, *15*, 577.
- [18] Liu, G.; Walsh, A. G.; Zhang, P. Synergism of iron and platinum species for low-temperature CO oxidation: From two-dimensional surface to nanoparticle and single-atom catalysts. *J. Phys. Chem. Lett.* **2020**, *11*, 2219–2229.
- [19] Ro, I.; Aragao, I. B.; Chada, J. P.; Liu, Y. F.; Rivera-Dones, K. R.; Ball, M. R.; Zanchet, D.; Dumesic, J. A.; Huber, G. W. The role of Pt–Fe<sub>2</sub>O<sub>3</sub> interfacial sites for CO oxidation. *J. Catal.* **2018**, *358*, 19–26.
- [20] Song, S. F.; Wu, Y. J.; Ge, S. S.; Wang, L.; Wang, Y. S.; Guo, Y. L.; Zhan, W. C.; Guo, Y. A facile way to improve Pt atom efficiency for CO oxidation at low temperature: Modification by transition metal oxides. *ACS Catal.* **2019**, *9*, 6177–6187.
- [21] Liu, Z.; Liu, K. J.; Yang, X.; Chen, X. H.; Shen, X.; Li, Y. N.; Fang, Y. F.; Liu, Y. J.; Zhao, J. W.; Yang, X. G. et al. *In-situ* formed stable Pt nanoclusters on ceria-zirconia solid solutions induced by hydrothermal aging for efficient low-temperature CO oxidation. *Chem. Eng. J.* **2024**, *498*, 155427.
- [22] Gaio, C. C.; Pinto, J. C.; Schmal, M. Effects of metal promoters (M = Fe, Co, and Cu) in Pt/M<sub>2</sub>Zr<sub>2</sub>O<sub>7</sub> catalysts and influence of CO<sub>2</sub> and H<sub>2</sub>O on the CO oxidation activity (PROX): Analysis of surface properties after reaction. *ACS Omega* **2024**, *9*, 25715–25729.
- [23] Qiao, B. T.; Wang, A. Q.; Yang, X. F.; Allard, L. F.; Jiang, Z.; Cui, Y. T.; Liu, J. Y.; Li, J.; Zhang, T. Single-atom catalysis of CO oxidation using Pt<sub>1</sub>/FeO<sub>x</sub>. *Nat. Chem.* **2011**, *3*, 634–641.
- [24] Chen, G. X.; Zhao, Y.; Fu, G.; Duchesne, P. N.; Gu, L.; Zheng, Y. P.; Weng, X. F.; Chen, M. S.; Zhang, P.; Pao, C. W. et al. Interfacial effects in iron-nickel hydroxide-platinum nanoparticles enhance catalytic oxidation. *Science* **2014**, *344*, 495–499.
- [25] Fu, Q.; Li, W. X.; Yao, Y. X.; Liu, H. Y.; Su, H. Y.; Ma, D.; Gu, X. K.; Chen, L. M.; Wang, Z.; Zhang, H. et al. Interface-confined ferrous centers for catalytic oxidation. *Science* **2010**, *328*, 1141–1144.
- [26] Lee, S.; Kim, S.; Kim, T.; Lin, C.; Lee, W. J.; Kim, S. J.; Gorte, R. J.; Jung, W. The effects of iron oxide overlayers on Pt for CO oxidation. *Catal. Commun.* **2022**, *172*, 106549.
- [27] Siani, A.; Alexeev, O. S.; Lafaye, G.; Amiridis, M. D. The effect of Fe on SiO<sub>2</sub>-supported Pt catalysts: Structure, chemisorptive, and catalytic properties. *J. Catal.* **2009**, *266*, 26–38.
- [28] Qi, Y. N.; Chen, H. Q.; Hong, F.; Cai, X. B.; Ying, Z. H.; Diao, J. Y.; Jia, Z. M.; Chen, J. W.; Wang, N.; Xiang, S. L. et al. Atomically dispersed Pt–Ru dual-atom catalysts for efficient low-temperature CO oxidation reaction. *Nano-Micro Lett.* **2026**, *18*, 172.
- [29] Tramontano, C.; Chianese, G.; Terracciano, M.; de Stefano, L.; Rea, I. Nanostructured biosilica of diatoms: From water world to biomedical applications. *Appl. Sci.* **2020**, *10*, 6811.
- [30] Lopez, P. J.; Desclès, J.; Allen, A. E.; Bowler, C. Prospects in diatom research. *Curr. Opin. Biotechnol.* **2005**, *16*, 180–186.
- [31] Fischer, C.; Adam, M.; Mueller, A. C.; Sperling, E.; Wustmann, M.; van Pée, K. H.; Kaskel, S.; Brunner, E. Gold nanoparticle-decorated diatom biosilica: A favorable catalyst for the oxidation of D-glucose. *ACS Omega* **2016**, *1*, 1253–1261.
- [32] Liang, H.; Zhou, S.; Chen, Y. T.; Zhou, F.; Yan, C. J. Diatomite coated with Fe<sub>2</sub>O<sub>3</sub> as an efficient heterogeneous catalyst for degradation of organic pollutant. *J. Taiwan Inst. Chem. Eng.* **2015**, *49*, 105–112.
- [33] Li, Q.; Zhai, G. Z.; Xu, Y.; Odoom-Wubah, T.; Jia, L. S.; Huang, J. L.; Sun, D. H.; Li, Q. B. Diatomite supported Pt nanoparticles as efficient catalyst for benzene removal. *Ind. Eng. Chem. Res.* **2019**, *58*, 14008–14015.
- [34] Vrieling, E. G.; Sun, Q. Y.; Beelen, T. P. M.; Hazelaar, S.; Gieskes, W. W. C.; Van Santen, R. A.; Sommerdijk, N. A. J. M. Controlled silica synthesis inspired by diatom silicon biomineralization. *J. Nanosci. Nanotechnol.* **2005**, *5*, 68–78.
- [35] Reid, A.; Buchanan, F.; Julius, M.; Walsh, P. J. A review on diatom biosilicification and their adaptive ability to uptake other metals into their frustules for potential application in bone repair. *J. Mater. Chem. B* **2021**, *9*, 6728–6737.
- [36] De Tommasi, E.; Gielis, J.; Rogato, A. Diatom frustule morphogenesis and function: A multidisciplinary survey. *Mar. Genomics* **2017**, *35*, 1–18.
- [37] Zhang, Z. H.; Wang, Z. Y. Diatomite-supported Pd nanoparticles: An efficient catalyst for Heck and Suzuki reactions. *J. Org. Chem.* **2006**, *71*, 7485–7487.
- [38] Jantschke, A.; Herrmann, A. K.; Lesnyak, V.; Eychmüller, A.; Brunner, E. Decoration of diatom biosilica with noble metal and semiconductor nanoparticles (< 10 nm): Assembly, characterization, and applications. *Chem. Asian J.* **2012**, *7*, 85–90.
- [39] Dehestaniathar, S.; Khajelakzay, M.; Ramezani-Farani, M.; Ijadpanah-Saravi, H. Modified diatomite-supported CuO–TiO<sub>2</sub> composite: Preparation, characterization and catalytic CO oxidation. *J. Taiwan Inst. Chem. Eng.* **2016**, *58*, 252–258.
- [40] Bahramian, B.; Doulati Ardejani, F.; Mirkhani, V.; Badii, K. Diatomite-supported manganese Schiff base: An efficient catalyst for oxidation of hydrocarbons. *Appl. Catal. A: Gen.* **2008**, *345*, 97–103.
- [41] Darouich, O.; Baaziz, W.; Ihiawakrim, D.; Hirlmann, C.; Spehner, D.; Schultz, P.; Poncet, H.; Rouchon, V.; Labidi, S.; Petit, C. et al. 3D multiscale analysis of the hierarchical porosity in *Coscinodiscus* sp. diatoms using a combination of tomographic techniques. *Nanoscale Adv.* **2022**, *4*, 1587–1598.
- [42] Benzemat, L. A.; Cherraka, R.; Hadjel, M.; Ketteb, A.; Goual, N. Characterization between crude diatomite and diatomite treated chemically. *Alger. J. Environ. Sci. Technol.* **2019**, *5*, 1107–1112.
- [43] Inchaurren, N.; Font, J.; Ramos, C. P.; Haure, P. Natural diatomites: Efficient green catalyst for Fenton-like oxidation of Orange II. *Appl. Catal. B: Environ.* **2016**, *181*, 481–494.
- [44] Sall, S.; Papaefthimiou, V.; Dintzer, T.; Vigneron, F.; Brendlé, E.; Petit, P.; Ferte, T.; Haddoum, S.; Labidi, S.; Petit, C. Diatom and diatomite: Different focus on natural media to material science path. *Am. J. Anal. Chem.* **2024**, *15*, 1–29.
- [45] Losic, D.; Rosengarten, G.; Mitchell, J. G.; Voelcker, N. H. Pore architecture of diatom frustules: Potential nanostructured membranes for molecular and particle separations. *J. Nanosci. Nanotechnol.* **2006**, *6*, 982–989.
- [46] Salameh, S. I. Y.; Khalili, F. I.; Al-Dujaili, A. H. Removal of U(VI) and Th(IV) from aqueous solutions by organically modified diatomaceous earth: Evaluation of equilibrium, kinetic and thermodynamic data. *Int. J. Miner. Process.* **2017**, *168*, 9–18.
- [47] Zhang, G. L.; Cai, D. Q.; Wang, M.; Zhang, C. L.; Zhang, J.; Wu, Z. Y. Microstructural modification of diatomite by acid treatment, high-speed shear, and ultrasound. *Microporous Mesoporous Mater.* **2013**, *165*, 106–112.
- [48] Kogel, J. E.; Trivedi, N. C.; Barker, J. M.; Krukowski, S. T. *Industrial Minerals & Rocks: Commodities, Markets, and Uses*, 7th ed.; Society for Mining, Metallurgy, and Exploration: Littleton, 2006.

- [49] Raven, J. A. Iron in diatoms. In *Diatoms: Fundamentals and Applications*; Seckbach, J.; Gordon, R., Eds.; Wiley: Hoboken, 2019; pp 213–224.
- [50] Al-Samarrai, K. Chemical, physical and mineralogical characterization of Al-Hishal Diatomite at Subkhah Ghuzayil area Libya. *Int. J. Res. Appl. Nat. Soc. Sci.* **2014**, *2*, 165–174.
- [51] Gao, X.; Bowler, C.; Kazamia, E. Iron metabolism strategies in diatoms. *J. Exp. Bot.* **2021**, *72*, 2165–2180.
- [52] Xu, Y.; Axe, L. Synthesis and characterization of iron oxide-coated silica and its effect on metal adsorption. *J. Colloid Interface Sci.* **2005**, *282*, 11–19.
- [53] Mosallanejad, S.; Dlugogorski, B. Z.; Kennedy, E. M.; Stockenhuber, M. On the chemistry of iron oxide supported on  $\gamma$ -alumina and silica catalysts. *ACS Omega* **2018**, *3*, 5362–5374.
- [54] Tomatis, M.; Xu, H. H.; Wei, C. H.; Bishop, M. T.; He, J.; Wang, C. J.; Zhao, M.; Xiao, H.; Yu, H.; Behera, S. N. et al. A comparative study of Mn/Co binary metal catalysts supported on two commercial diatomaceous earths for oxidation of benzene. *Catalysts* **2018**, *8*, 111.
- [55] Plodinec, M.; Nerl, H.; Schlögl, R.; Lunkenbein, T. Direct insight into the reactivity of Pt nanoparticles in CO oxidation by operando TEM and the impact of electron dose rate on their coarsening. *Microsc. Microanal.* **2020**, *26*, 1868–1871.
- [56] Przybek, A.; Łach, M.; Hebdowska-Krupa, M.; Miernik, K.; Mikula, J. Research of the physical and chemical properties of diatomite as a carrier of phase change materials for use in advanced building materials. *Front. Mater.* **2024**, *11*, 1507779.
- [57] Ibrahim, S. S.; Selim, A. Q. Heat treatment of natural diatomite. *Physicochem. Probl. Miner. Process.* **2012**, *48*, 413–424.
- [58] Ivanova, S.; Pitchon, V.; Petit, C.; Caps, V. Support effects in the gold-catalyzed preferential oxidation of CO. *ChemCatChem* **2010**, *2*, 556–563.
- [59] Chakraborty, A. R.; Toma, F. T. Z.; Alam, K.; Yousuf, S. B.; Hossain, K. S. RETRACTED: Influence of annealing temperature on  $\text{Fe}_2\text{O}_3$  nanoparticles: Synthesis optimization and structural, optical, morphological, and magnetic properties characterization for advanced technological applications. *Heliyon* **2024**, *10*, e40000.



This is an open access article under the terms of the Creative Commons Attribution 4.0 International License (CC BY 4.0, <https://creativecommons.org/licenses/by/4.0/>).

© The Author(s) 2026. Published by Tsinghua University Press.



清华大学出版社  
Tsinghua University Press

SciOpen