Supplementary Information

Surface properties and thermal stability of SiO₂-crystalline TiO₂ nanocomposites

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Synthesis procedure

Synthesis of the host support (SBA, Table 1)

The SBA-15 support is prepared according to the procedure proposed by Roggenbuck et al. A mass of 12.0 g of triblock copolymer P123 is dissolved in 360 g of distilled water and 43 g of HCl 32 wt.%. The solution is heated at 35 °C. After 1 hour to ensure the complete dissolution of the copolymer, 24 g of TEOS is slowly added under vigorous stirring, given a synthesis composition 1 TEOS : 0.018 P123 : 3.3 HCl : 187 H2O (molar ratio). The transparent solution is stirred at 35 °C for 24 h, and the milky solution obtained is transferred in a Teflon-line autoclave for treatment at 140 °C for 24 h. The autoclave is cooled down to room temperature, and the white solid filtered, washed with distilled water, and dried at 80 °C overnight. Before use and characterization, the solid is calcined under air at 550 °C for 3 h (temperature increase ramp = 1 °C.min⁻¹).

Characterization

X-Ray Diffraction

The Scherrer equation used after Warren’s correction for instrumental broadening is:

\[ D = \frac{K\lambda}{\beta \cos \theta} \]

K is a constant assumed equal to 0.86 and \( \lambda \) is the wavelength of the X-ray source. \( \beta \) is the effective linewidth of the X-ray reflection, calculated by the formula \( \beta^2 = B^2 - b^2 \) where B is the full width at half maximum (FWHM) of the reflection and b is the instrumental broadening determined by the FWHM of X-ray reflection of quartz having particles larger than 150 nm.

IR spectroscopy

The silanol (SiOH) stretching band surface is deduced from spectra decomposition, using the peak solve fitting from OMNIC. Peak type is fixed to a Gaussian/Lorentzian function centered at 3745 ± 5 cm⁻¹, with a maximum full width at half height (FWHM) of 15 cm⁻¹. Residual TiOH area is estimated by subtraction of the SiOH area to the total free OH area, assuming contributions of SiOH and TiOH as purely additive. The determination of the OH surface density is calculated using the Beer-Lambert law:

\[ n(\text{OH}) = \frac{A \times S \times N_A}{\varepsilon \times S_{\text{BET}} \times m} \]

Where \( \varepsilon \) is the molar absorption coefficient for the SiOH species, equal to 1 cm.mol⁻¹ at 3750 cm⁻¹, \( n(\text{OH}) \) is the OH surface density (OH.nm⁻²), A is the IR absorbance (cm⁻¹), S is the surface of the self-supported wafer (cm²), \( N_A \) is the Avogadro’s number, \( S_{\text{BET}} \) is the specific surface area (m².g⁻¹), and m the weight of the wafer (g).

The oxygen isotopic exchange (OIE) reaction

Experiment is performed from 400°C to 800°C (ramp =5 °C.min⁻¹), and partial pressure evolution of \( ^{18}\text{O}_2 \) (mass 36), \( ^{16}\text{O}_2 \) (mass 32), and \( ^{16}\text{O}^{18}\text{O} \) (mass 34) are recorded on a mass spectrometer allowing quantification of the \(^{18}\text{O} \) fraction in the gas phase (\( \alpha \)), the number of oxygen atom exchanged (\( N_{\text{ex}} \)), and the fraction of the solid exchanged at the end of the experiment (\( \text{O}_{\text{ex}} \)).

\( \alpha \) is calculated from the partial pressure of \(^{18}\text{O}_2\), \(^{16}\text{O}_2\) and \(^{18}\text{O}^{16}\text{O} \) at each time :

\[ \alpha = \frac{\frac{1}{2} \left( P^{({18}\text{O}^{16}\text{O})} + P^{({18}\text{O}_2)}) \right)}{P^{({16}\text{O}_2)} + P^{({18}\text{O}^{16}\text{O})} + P^{({18}\text{O}_2)})} \]
N_{ex} is calculated from the $^{18}$O at the end of the reaction:

$$N_{ex} = (100 - \alpha) \times N_g$$

Where $N_g =$ total number of oxygen atoms in the gas phase.

$O_{ex}$ is obtained by the following formula:

$$O_{ex} = \frac{100 - \alpha}{100 - \%^{(18)O}} \cdot 100$$

Figure S1. Evolution of the N\textsubscript{2} adsorption – desorption isotherms (A) and NLDFT pore size distributions (B) with the titania loading in the nanocomposites. Isotherms are each offset from 600 cm\textsuperscript{3}.g\textsuperscript{-1} (with horizontal bars added in the figure to identify the offset).
**Figure S2.** Evolution of the small-angle (A) and wide-angle (B) X-ray diffraction patterns with titania loading in the nanocomposites.
Figure S3. Evolution of the main physical properties of the nanocomposites with the titania loading after calcination at 400 °C.
Figure S4. High magnification TEM images obtained over 20Ti-SBA (A) and 55Ti-SBA (B) after calcination at 800 °C.
**Figure S5.** Evolution of the Raman spectra with the calcination temperature of the composites. (A) 20Ti-SBA; (B) 55Ti-SBA.