

Embedding catalytic nanoparticles inside mesoporous structures with controlled porosity: Au@TiO₂

Electronic Supplementary Information

Raquel Nafria,^a Pilar Ramírez de la Piscina,^b Narcís Homs,^{a,b} Joan Ramon Morante,^{a,c} Andreu Cabot,^{*,a,c} Urbano Diaz,^d Avelino Corma^{*,d}

^a Catalonia Institute for Energy Research, IREC, Sant Adrià del Besos, Barcelona, 08930, Spain

^b Departament de Química Inorgànica, Universitat de Barcelona, Barcelona, 08028, Spain

^c Departament d'Electrònica, Universitat de Barcelona, Barcelona, 08028, Spain

^d Instituto de Tecnología Química (UPV-CSIC), Universidad Politécnica de Valencia, Avenida de los Naranjos s/n 46022 Valencia, Spain

* Emails: acabot@irec.cat ; acorma@itq.upv.es

Table SII. Textural properties of inorganic-organic hybrid materials (before calcination).

As-prepared	Surface area (m ² /g)			Pore volume (cm ³ /g)	
	<i>S_{BET}</i>	<i>Microporous</i>	<i>Mesoporous</i>	<i>V_{tot}</i>	<i>VB_{JH}</i>
TiO ₂	552	496	56	0.34	
TiO ₂ (t)	226	103	123	0.86	0.82
TiO ₂ (n)	176	75	101	0.61	0.59
TiO ₂ (b)	341	140	201	1.21	1.22
Au@TiO ₂	349	10	254	0.32	0.27
Au@TiO ₂ (t)	293	87	206	1.86	1.83
Au@TiO ₂ (n)	183	36	147	0.76	0.75
Au@TiO ₂ (b)	341	75	239	1.32	1.21

Table SI2. Textural properties of the calcined material.

Calcined	Surface area (m ² /g)			Pore volume (cm ³ /g)	
	<i>S_{BET}</i>	<i>Microporous</i>	<i>Mesoporous</i>	<i>V_{tot}</i>	<i>VB_{JH}</i>
TiO ₂	64	64	-	0.11	
TiO ₂ (t)	85	42	43	0.37	0.39
TiO ₂ (n)	153	47	106	0.59	0.56
TiO ₂ (b)	180	54	126	0.84	0.82
Au@TiO ₂	89	8	81	0.16	0.16
Au@TiO ₂ (t)	109	12	97	0.57	0.59
Au@TiO ₂ (n)	178	44	134	0.74	0.72
Au@TiO ₂ (b)	145	53	91	0.82	0.80

Table SI3 . ICP Au content (% wt) of the calcined materials.

Sample name	[Au]
Au@TiO ₂	0.50
Au@TiO ₂ (t)	1.08
Au@TiO ₂ (n)	0.91
Au@TiO ₂ (b)	0.95
Au/TiO ₂ (b)	1.48

X-ray photoelectron spectroscopy analysis

X-ray photoelectron spectroscopy (XPS) analysis was used to characterize the surface composition and chemical state of Au@TiO₂ materials (Figure SI1). XPS was performed with a PHI 5500 Multitechnique System, using a monochromatic Al K α X-ray source (1486.6 eV, 350 W) which was placed perpendicular to the analyzer. Acquisition time was 2 min for the C 1s, Ti 2p and O1s regions and 60 min for Au 4f. The binding energy scale was corrected for surface charging by taking the C 1s peak of contaminant carbon as a reference at 284.8 eV

From the XPS analysis the Au/Ti ratio was calculated to be on the order of 1×10^{-4} , pointing out the embedment of gold within the TiO₂ matrix

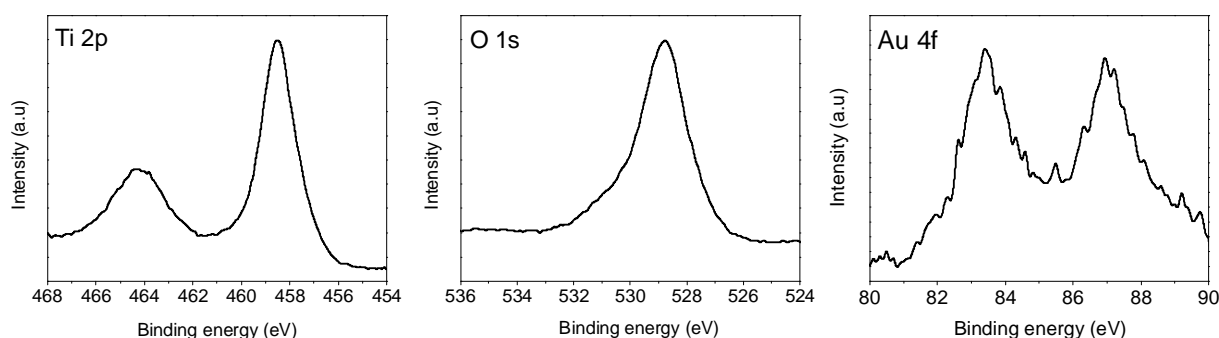


Figure SI1. XPS spectrum of Au@TiO₂(b)

Thermogravimetry analysis

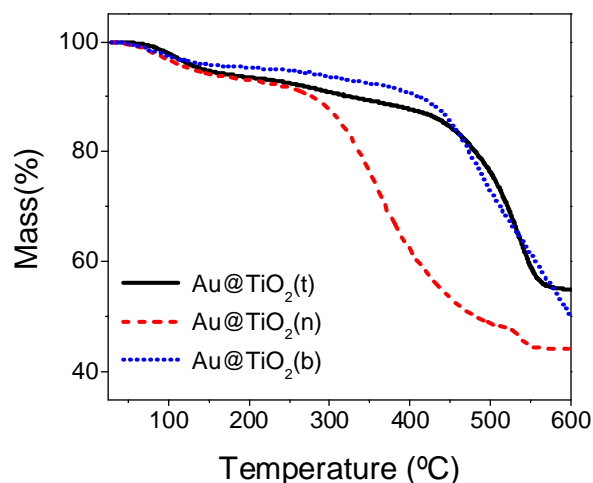


Figure SI2. Thermogravimetric profile of the Au@TiO₂ mesoporous materials.

Scanning electron microscopy (SEM)

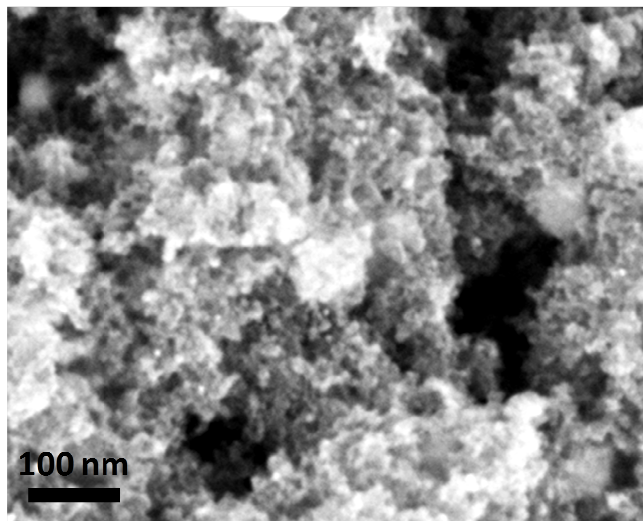


Figure SI3. SEM micrograph of the Au@TiO₂ (b) mesoporous materials.

Selected Area Electron Diffraction pattern

The gold crystallographic planes (002) and (222) were detected by electron diffraction inside the TEM.

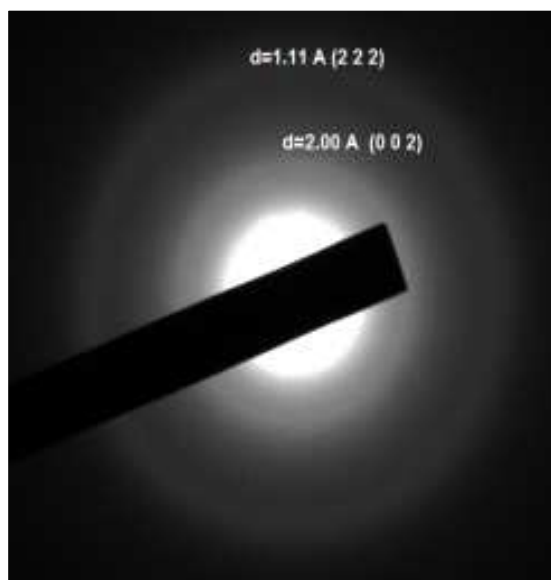


Figure SI4. SAED pattern of the calcined Au@TiO₂ (n) material.