

## Microwave-Assisted Flow Synthesis of Nano-confined Pt in Titania Nanotubes (Pt-TiNT) photocatalysts

**Miguel A. Bañares**<sup>1</sup>, Yingjian Luo<sup>2</sup>, **Ana Serrano-Lotina**<sup>1</sup>, Felicia Febriana Budihardjo<sup>3</sup>, Shabnam Taghipur<sup>3</sup>, Shammi Akter Ferdousi<sup>3</sup>, Liping Li<sup>2</sup>, Juan Jose Delgado<sup>4</sup>, Ángel López-Buendía<sup>5</sup>, Raquel Portela<sup>2</sup>, Wei Han<sup>2,6</sup>, King Lun Yeung<sup>2</sup>

<sup>1</sup>Spectroscopy and Industrial Catalysis, Instituto de Catálisis y Petroleoquímica, CSIC-ICP, Marie Curie 2, E-28049-Madrid, Spain

<sup>2</sup>Division of Environment and Sustainability, The Hong Kong University of Science and Technology, Clear Water Bay, Kowloon, Hong Kong SAR.

<sup>3</sup>Department of Chemical and Biological Engineering, The Hong Kong University of Science and Technology, Clear Water Bay, Kowloon, Hong Kong SAR.

<sup>4</sup>Department of Materials Sciences and Metallurgical Engineering and Inorganic Chemistry Universidad de Cadiz, Campus Universitario de Puerto Real 11519 Puerto Real, Cádiz, Spain

<sup>5</sup>CEINNMAT, SL, Catedrático Agustín Escardino 9, E-46980-Paterna, Valencia, Spain

<sup>6</sup>HKUST Shenzhen-Hong Kong Collaborative Innovation Research Institute, Futian, Shenzhen, Guangdong, China

[miguel.banares@csic.es](mailto:miguel.banares@csic.es); [asl@icp.csic.es](mailto:asl@icp.csic.es)

Pt-TiNT with PtO nanoparticles dispersed within the lumen and interlayer spaces of titania nanotubes (TiNT) were prepared by a new process involving titania nanosheets (TiNS) synthesis in an optimised microwave-assisted flow reactor, followed by ion-exchange with a Pt precursor, before triggering the titanate layer rolling to trap the Pt precursor clusters inside the titanium nanotubes, and thermal treatment. TEM, XRD and Raman analyses confirm the total conversion of TiO<sub>2</sub> into TiNS in 15 min at 120°C and 4 bar, and TiNS transformation into 181 nm-long TiNT with 10 and 6 nm outer and inner diameter, respectively. The 2% Pt-TiNT comprises PtO clusters (according to XPS) of 0.7 nm diameter, causing slight distortions of the interlayer spaces while some larger 2-3 nm Pt clusters reside within the lumen. Pt-TiNT is 14-fold more active than TiNT for visible light photocatalytic oxidation of diclofenac and more than 1000-fold better than the uncatalyzed photoconversion reaction. Nano-confinement of PtO clusters narrowed the bandgap of the TiNT, which combined with its excellent absorptivity to harvest light allowed a large spectral range of photon energies to activate the photocatalyst.

This work is supported by the Horizon 2020 BIORIMA project and the Hong Kong Research Grant Council E-HKUST601/17 and in part by the Project of Hetao Shenzhen-Hong Kong University of Science and Technology Innovation Cooperation Zone (HZQB-KCZYB-2020083). Dr. Y.J. Luo stay at the Instituto de Catálisis y Petroleoquímica is supported by the HKUST Overseas Research Award. Finally, the authors acknowledge the support of the Central Facilities of the Hong Kong University of Science and Technology including the Material Characterization and Preparation Facility (MCPF) and the Environmental Central Facility (ENVF).