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Controlling Anatase and Rutile Polymorph Selectivity During Low-Temperature Synthesis of Mesoporous TiO₂ Films

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S1. Thermogravimetric analysis.

All TGA measurements were performed in air using a Perkin Elmer TGA 7 Thermogravimetric Analyser, using a 20 ml/min air flow and a heating rate of 2 °C/min.

TGA measurements of samples A-E after washing are shown in Figure S1. An initial mass loss between 7 wt% and 10 wt% are observed up to a temperature of 150 °C and is attributed to loss of water adsorbed on the surface of the titanium dioxide nanoparticles. A second region of mass loss between 6 wt% and 10 wt% with an onset at approximately 200 °C are clearly visible for samples A-E and is attributed to the loss of organic materials adsorbed onto the surfaces of the nanoparticles. Considering that the initial weight ratio of the organic block co-polymer Pluronic F127 to the theoretically maximum yield of titania is 2.25:0.35 in the reaction solution, the TGA results shows that the performed washing protocol removes 98-99% of the organic materials initial present during the synthesis.



Figure S1. TGA curves for samples A, B, C, D and E reacted for 20h, 40h, 96h, 170h and 336h, respectively. The mass loss region between 25 °C and 150 °C is attributed to surface adsorbed water. The onset of mass loss observed at approximately 200 °C is attributed to loss of organic material adsorbed to the titanium dioxide nanoparticles surfaces.

TGA curves measured on films F, G and H after UV/ozone treatment are shown in Figure S2. As for samples A-E a mass loss region attributed to loss of adsorbed water is observed between 25 °C and approximately 150 °C for films F, G and H. However, the region above 200 °C associated with the loss of organic matter contains more features indicating a more diverse composition of organic materials adsorbed onto the surface of sample F, G and H. This could be explained by the UV/ozone cleaning procedure, possibly resulting in a distribution of partly photooxidized organic compounds. The total organic content is approximately 10 wt% for all films. As for samples A-E washed with solvent, the UV/ozone treatment of the prepared films F, G and H appears to remove most of the organic template.



Figure S2. TGA curves for sample films F, G and H aged for 100h, 200h and 300h, respectively. The mass loss region between 25 °C and 150 °C is attributed to surface adsorbed water. The onset of mass loss observed at approximately 200 °C is attributed to loss of organic material adsorbed to the titanium dioxide nanoparticle surfaces.

S2. Scanning electron microscopy (SEM)

SEM micrographs were collected using a LEO ULTRA 55 FEG from Leo Electron Microscopy operated at 5kV using an in-lens detector.

SEM micrographs of films F, G and H are shown in Figure S3 a, b and c, respectively. SEM micrographs of films F and G display wormhole-like features as well as pores in the mesorange. SEM micrographs of film H show a highly porous material with pore sizes in the mesorange.



Figure S3. SEM micrographs of films F (a), G (b), H (c) display pores within the mesorange.