

Supplementary Material

The Influence of Calcination Temperature on Photocatalytic Activity of TiO₂-Acetylacetonone Charge Transfer Complex Towards Degradation of NO_x under Visible Light

Lucas A. Almeida ¹, Margarita Habran ², Rafael dos Santos Carvalho ³,
Marcelo E. H. Maia da Costa ³, Marco Cremona ³, Bruno C. Silva ⁴, Klaus Krambrock ⁴,
Omar Ginoble Pandoli ⁵, Edisson Morgado Jr. ⁶ and Bojan A. Marinkovic ^{1,*}

¹ Department of Chemical and Materials Engineering, Pontifical Catholic University of Rio de Janeiro (PUC-Rio), 22453-900 Rio de Janeiro, RJ, Brazil; lucasalmeida@aluno.puc-rio.br

² Facultad de Ingeniería, Universidad ECCI, 111311 Bogotá, Colombia; nhabrane@ecci.edu.co

³ Department of Physics, Pontifical Catholic University of Rio de Janeiro (PUC-Rio), 22453-900 Rio de Janeiro, RJ, Brazil; rafael.santos@fis.puc-rio.br (R.d.S.C.); maiacosta@vdg.fis.puc-rio.br (M.E.H.M.d.C.); cremona@fis.puc-rio.br (M.C.)

⁴ Department of Physics, Federal University of Minas Gerais, 31270-901 Belo Horizonte, MG, Brazil; cordeiro@fisica.ufmg.br (B.C.S.); klaus@fisica.ufmg.br (K.K.)

⁵ Department of Chemistry, Pontifical Catholic University of Rio de Janeiro (PUC-Rio), 22453-900 Rio de Janeiro, RJ, Brazil; omarpandoli@puc-rio.br

⁶ PETROBRAS S.A., Research & Development Centre, 21941-915 Rio de Janeiro, RJ, Brazil; emorgado@petrobras.com.br

* Correspondence: bojan@puc-rio.br

Received: 12 November 2020; Accepted: 8 December 2020; Published: date

S1.

Fourier transform infrared (FTIR) spectrum of pure acetylacetonone

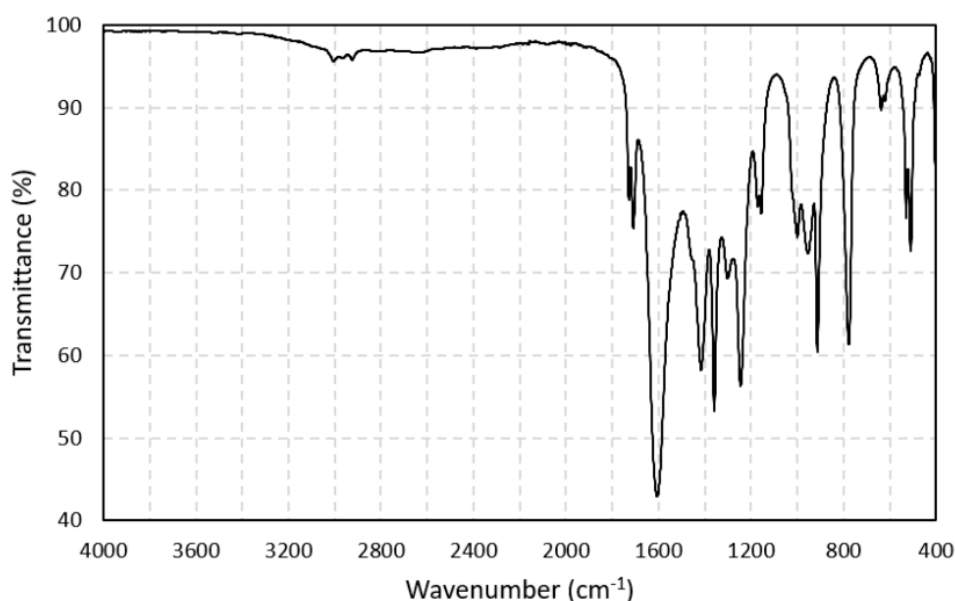


Figure S1. FTIR spectrum of acetylacetonone.

S2.

Fourier transform infrared (FTIR) spectra of TiO₂-ACAC, TiO₂-ACAC-300, TiO₂-ACAC-400

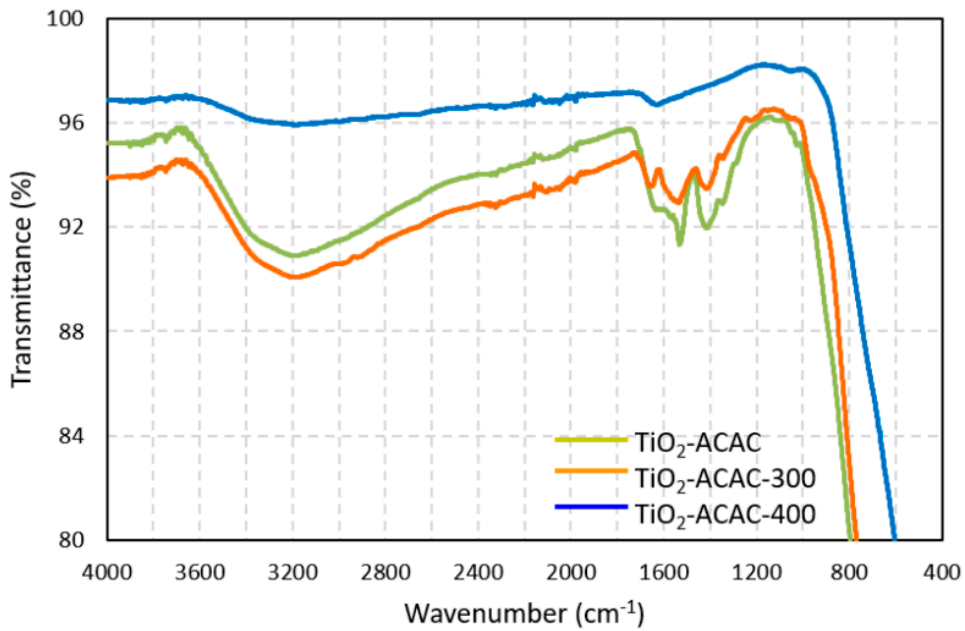


Figure S2. FTIR spectra of TiO₂-ACAC, TiO₂-ACAC-300 and TiO₂-ACAC-400.

S3.

N₂ adsorption-desorption isotherms of TiO₂-ACAC, TiO₂-ACAC-300 and TiO₂-ACAC-400

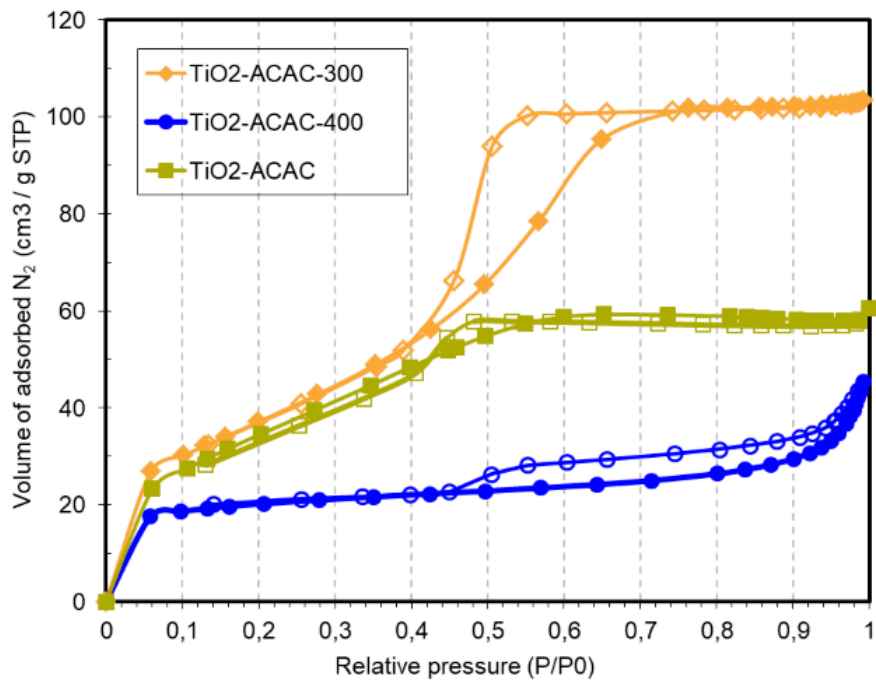


Figure S3. N₂ adsorption-desorption isotherms of TiO₂-ACAC, TiO₂-ACAC-300 and

TiO₂-ACAC-400.

T1.

Mean pore diameter of TiO₂-ACAC, TiO₂-ACAC-300 and TiO₂-ACAC-400

Table S1. Mean pore diameter.

Samples	Mean pore diameter (BJH), nm
TiO ₂ -ACAC	2.9
TiO ₂ -ACAC-300	4
TiO ₂ -ACAC-400	7.9

S4.

XPS spectra within Ti 2p range of TiO₂-ACAC, TiO₂-ACAC-300, TiO₂-ACAC-400 and TiO₂-ACAC-550

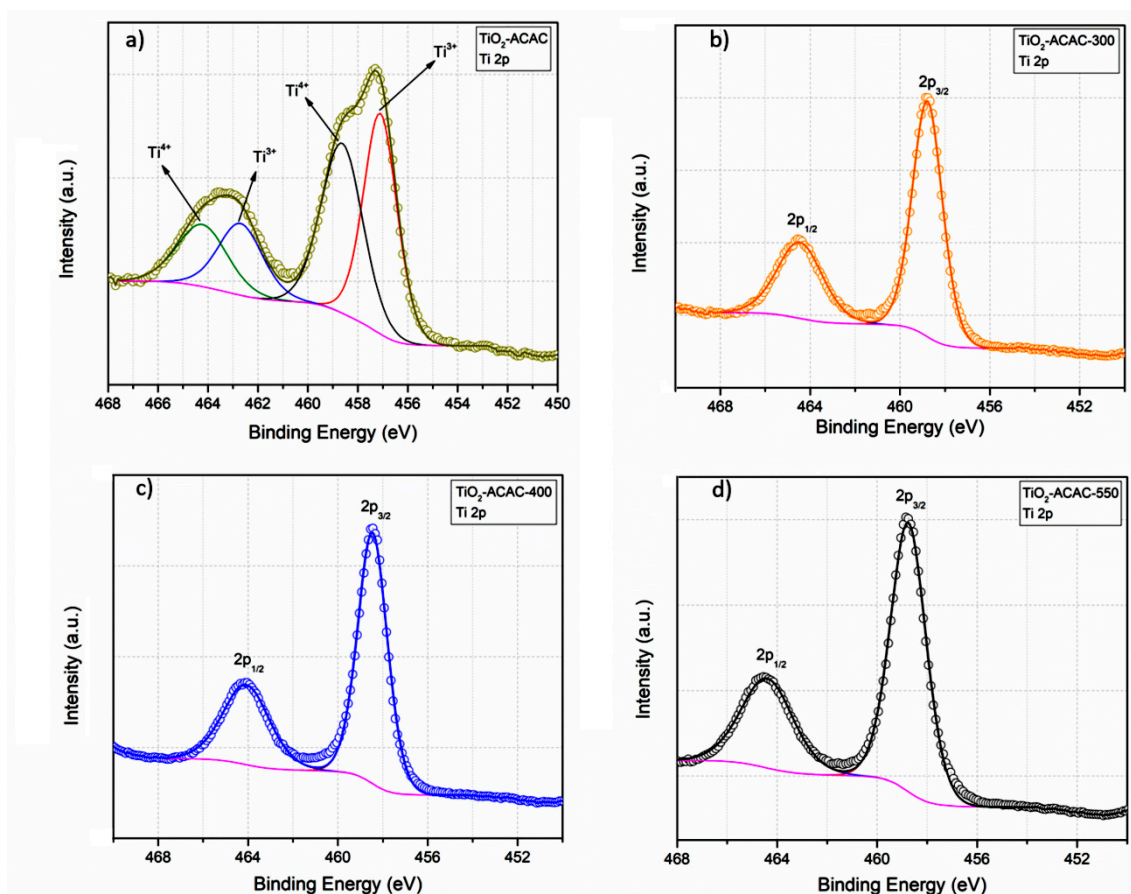


Figure S4. XPS spectra within Ti 2p range of TiO₂-ACAC, TiO₂-ACAC-300, TiO₂-ACAC-400 and TiO₂-ACAC-550.

S5.

XPS spectra within C 1s range of TiO₂-ACAC, TiO₂-ACAC-300, TiO₂-ACAC-400 and TiO₂-ACAC-550

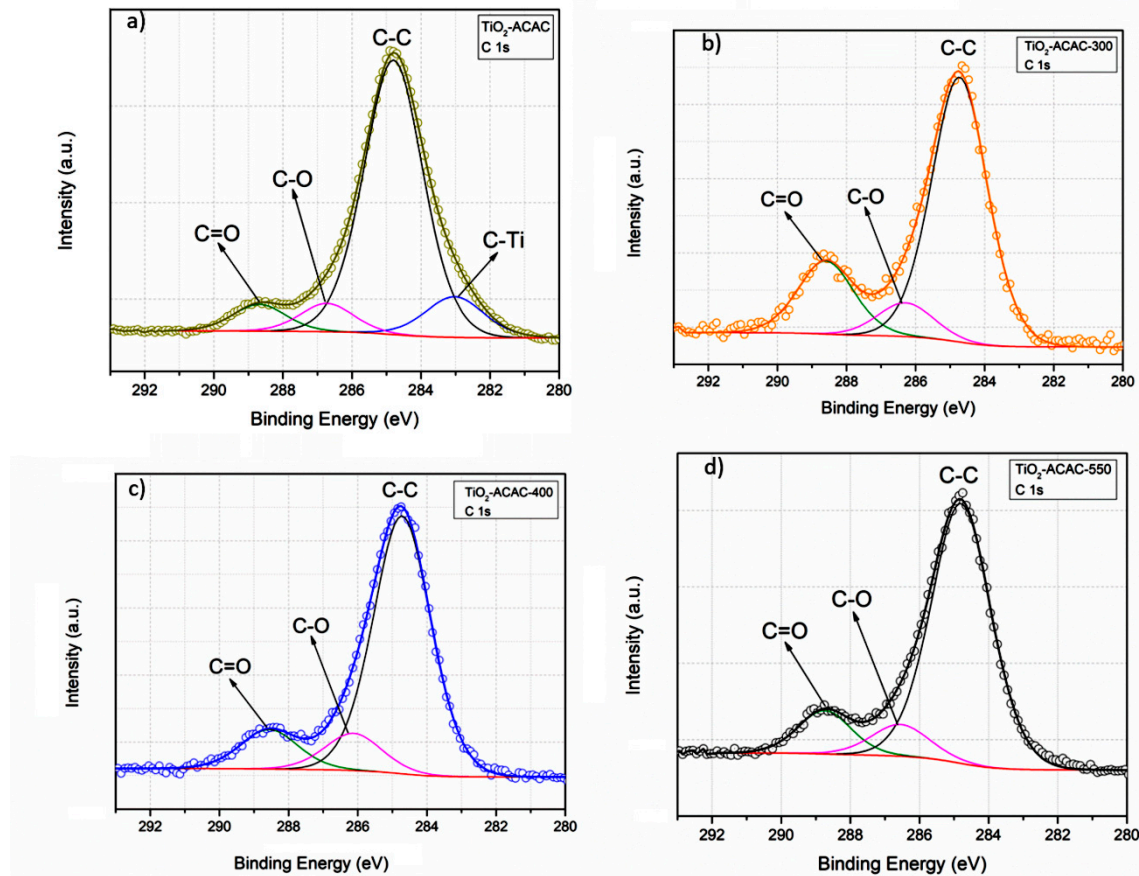


Figure S5: XPS spectra within C 1s range of TiO₂-ACAC, TiO₂-ACAC-300, TiO₂-ACAC-400 and Table 2. ACAC-550.

S6.

XPS spectra within O 1s range of TiO₂-ACAC-300, TiO₂-ACAC-400 and TiO₂-ACAC-550

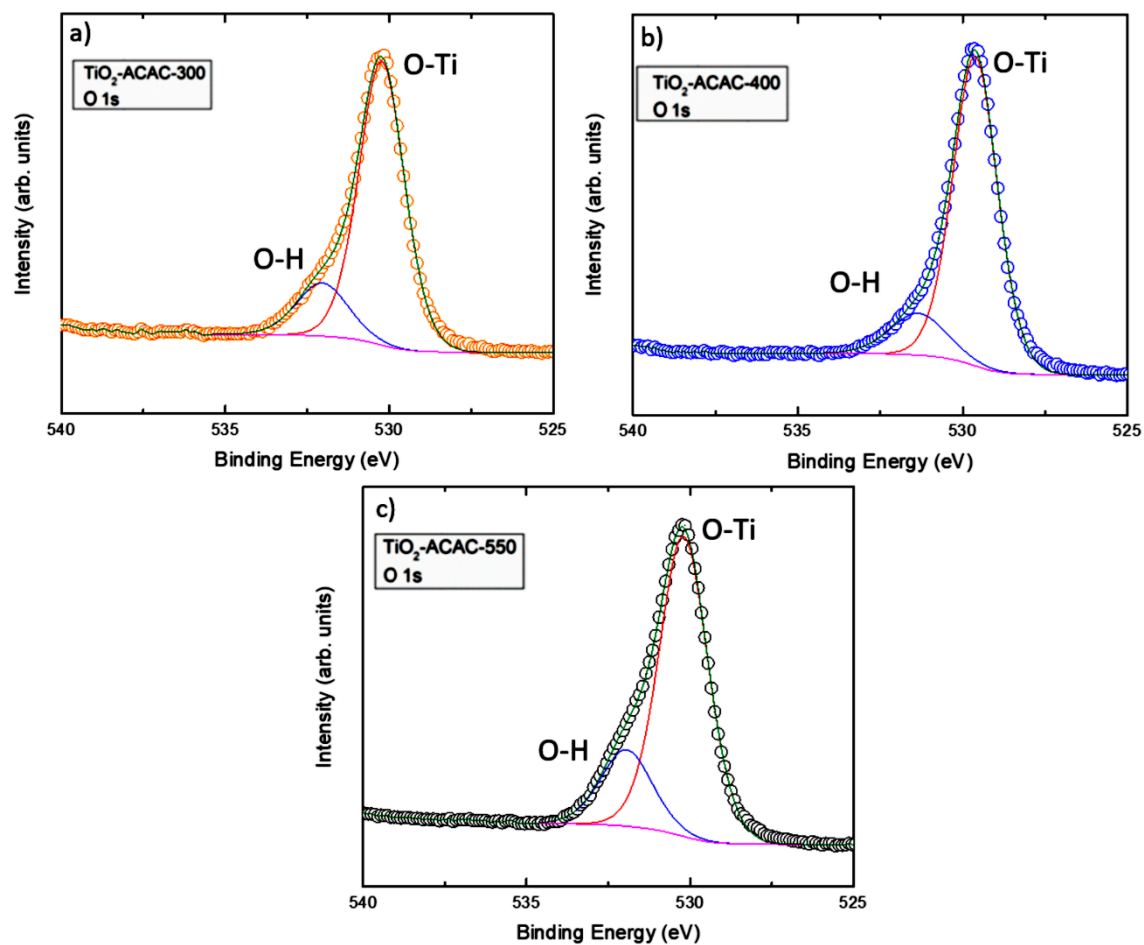


Figure S6. XPS spectra within O 1s range of TiO₂-ACAC-300, TiO₂-ACAC-400 and TiO₂-ACAC-550.

S7.

PL spectrum of TiO₂-ACAC-550

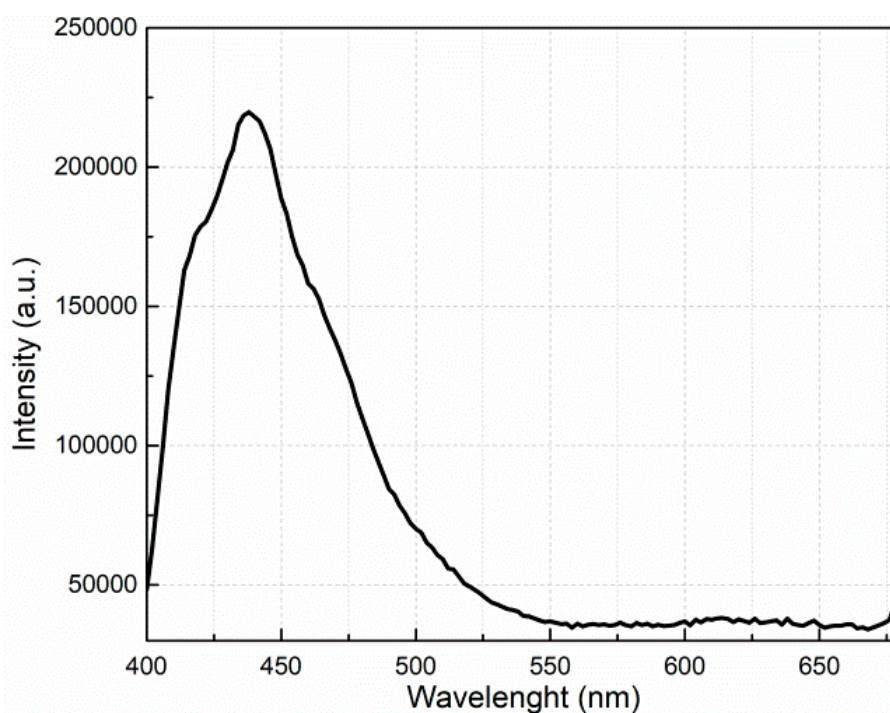


Figure S7. PL spectrum of TiO₂-ACAC-550.

S8.

Concentration of SETOV defects as a function of time for TiO₂-ACAC-300 and TiO₂-ACAC-400

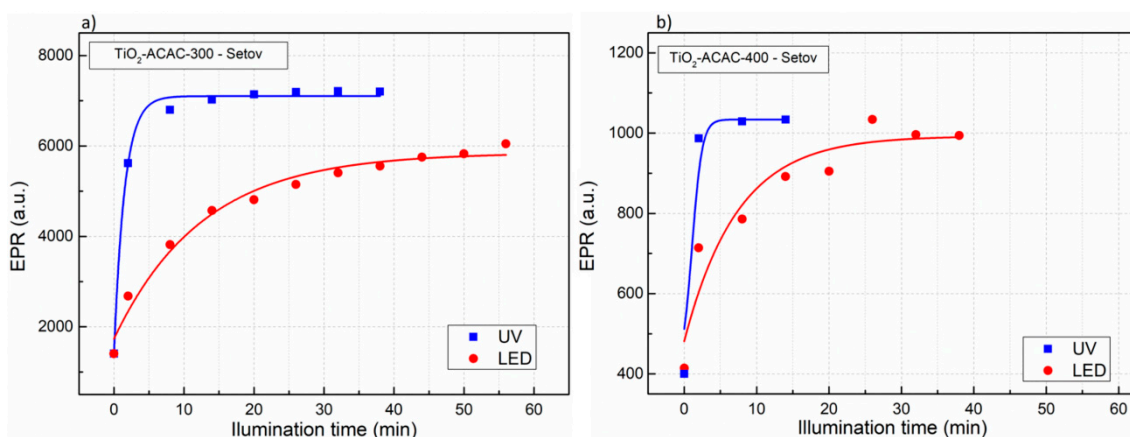


Figure S8. Concentration of SETOV defects as a function of time for TiO₂-ACAC-300 and TiO₂-ACAC-400, under visible (white LED) and ultraviolet radiation.

S9.

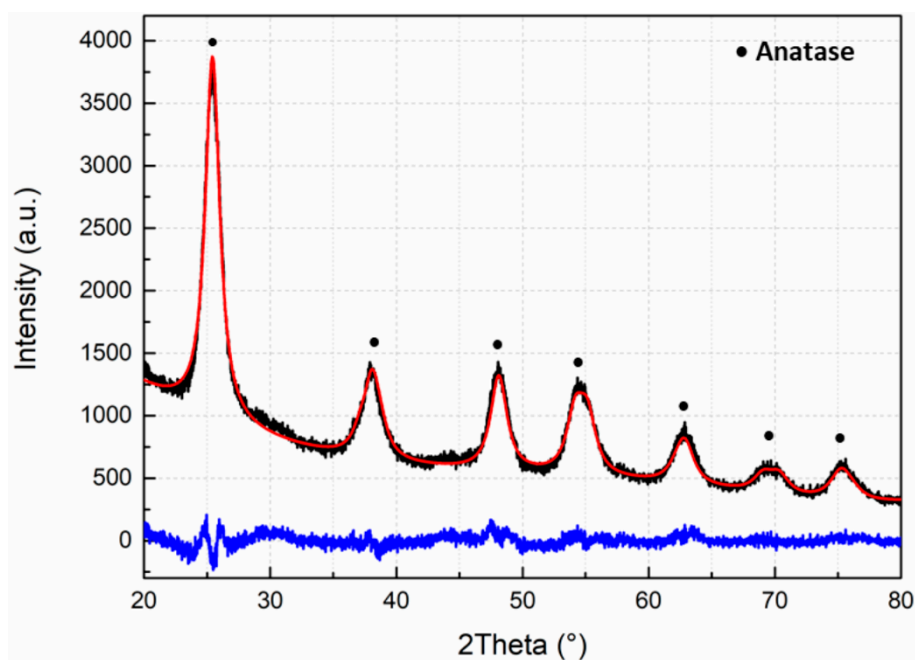
Experimental XRPD pattern of deactivated TiO₂-ACAC-300

Figure S9. Experimental XRPD patterns refined by Le Bail method for deactivated TiO₂-ACAC-300. The experimental pattern is black, the calculated pattern is red and the difference plot is blue.

S10.

Experimental procedure of Ion Chromatography analysis of virgin and deactivated photocatalysts

About 0.3 g of the deactivated powder was dispersed in 20 mL of distilled water and ultrasonicated for 30 min. Afterwards, the dispersion was filtered with filter paper with the aid of 5 mL of distilled water. The final volume of the filtered solution was 25 mL.

The same procedure was carried out with the virgin photocatalyst.

Ion Chromatograph Dionex-DX 2000 was used to measure content of ions in the as-prepared liquid, especially looking for NO₃⁻ since it was expected that this ion was adsorbed at the surface of deactivated photocatalysts.

For the measurements we used a precolumn AG 18 4 × 50 mm and the column AS 18 4 × 25 mm with the flux of solution of 1.0 mL min⁻¹.

The virgin powder presented 0.16 mg L⁻¹ of NO₃⁻, while the deactivated photocatalyst presented 6.40 mg L⁻¹ of NO₃⁻.