



Supplementary Information

Development and Characterization of Composite Carbon Adsorbents with Photocatalytic Regeneration Ability: Application to Diclofenac Removal from Water

Determination of ID/IG Ratio

Before the determination of the I_D/I_G ratio, the Raman spectra were normalized, and Gaussian fitting was done using the origin software. The integrated peak areas (for D and G bands) were used to calculate the I_D/I_G ratios of the different materials according to Figures S1–3.

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	Area	Standard error	
D band	21.22524	0.16694	
G band	2.21074	0.11382	
ID/ IG	9.60097		



Figure S1. Normalized Gaussian fitted D and G bands for HCP25.



Figure S2. Normalized Gaussian fitted D and G bands for HCP25W.



Figure S3. Normalized Gaussian fitted D and G bands for HCP25600.

Calculation of Anatase and Rutile Amounts from In Situ XRD Data at Different Temperatures

The Reference intensity ratio (RiR) method with corundum used as the internal reference and the scale factor defined by Intensity of the Analyte / Intensity of Corundum = I / Ic was used. The calculations were based on the peak intensities of Rutile and Anatase. The RiR column contains the ratios corrected by the RiR(I/Ic) parameters, which describes how well both phases scatter X-ray for standard sample mixtures. This method was equally used calculating the P25 anatase and rutile phase percentages for the conventional XRD results.

T[C]	Rutile [weight%]	Anatase [weight%]	error estimate[+/-%]
250	20.9	79.1	3.1
300	29.7	70.3	1.5
330	30.5	69.5	1.4
350	30.8	69.2	1.3
380	31.5	68.5	1.2
400	35.2	64.8	1.3
450	34.9	65.1	1.4

Table S1. Quantitative XRD using RiR method for in-situ data.



Figure S4. Quantified rutile amounts at different temperatures.

Table S2. Analysis is based on the following phase assignments:.

Phase name	Formula	Figure of merit	Phase reg. detail	DB card number
Rutile, syn	TiO ₂	0.750	ICDD (PDF-4+ 2021)	04-001-9262
anatase	TiO ₂	1.215	ICDD (PDF-4+ 2021)	04-021-3587

With example diffractogram:



Figure S5. Identification of crystalline phases.

Quantitative analysis is based on the assigned phases and the main peak intensities for rutile (peak 1 1 0), and anatase (peak 1 0 1) with their RiR values of 3.610 and 4.219 respectively. Rikagu PDXL2 analysis package has been used to process the data and obtain the quantitative results. Because of low count per second (cps) values from the in-situ measurement, careful corrections to background positions and main peaks has been applied before executing optimization of peak shapes and background.



Figure S6. Residual curve obtained after optimization of peak intensities with Split pseudo-Voigt as peak shape function and background optimization for critical region of main peaks used in RiR method.

Values of error come from PDXL2 estimation of standard deviation due to high noise of the datasets and resulting uncertainty of integrated intensities for peaks of identified phases.



Figure S7. Region of diffractogram with main peaks for each identified phase.



Figure S8. Amounts of anatase and rutile at different temperatures.

C1s, O1s, and Ti2p XPS Spectra for HCP25W



Below are the C1S, O1S and Ti2p xps spectra for HCP25W (not included in the main text because they are same as those of HC P25)

Figure 9. (a) C1S, (b) O1S, and (c) Ti2p XPS spectra for HC P25 W.

Summary of Peak Positions and Their Assignments for FTIR, XPS, and Raman Spectra

Wave number (cm ⁻¹)	Functional groups
641	Ti–O stretching
790	C–H bending for aromatic out of phase deformation
1450	C–O carboxyl group
2341, 2360	Gaseous CO ₂
2909, 2940	C-H alkane groups and aliphatic –CH2 and –CH3 stretching vibrations
3700	O-H vibrations of hydroxyl groups

Table 3. Peak assignments for Figure 5 (FTIR spectra of HCP25 and HCP25W).

Table S4. Peak assignments for Figure 4 (XPS of HCP25 and HCP25W).

Binding energy (eV)	Chemical bond
284.6, 285.5	C=C, CH _x , C–C
288.9	COO-
529.97	Ti–O (oxygen in TiO ₂)
531.09	С-ОН, С-О-С, СОО-
533.4	C=O
458.8	Ti2p _{3/2}
464.5	Ti2p _{1/2}
35.79	W4f _{7/2}
37.75	W4f _{5/2}

Table 5. Peak assignments for Figures 3a and 9a (Raman spectra modes for P25).

Structure	Raman frequency modes (cm ⁻¹)
	150 (Eg), 202 (Eg), 400 (B1g), 512 (A1g), 522 (B1g), 638 (Eg),
Anatase	796 is the first overtone of B_{1g} at 400.
Rutile	248 (Eg) , 442 (Eg).
	235, 320-360, 612 (A1g), 827 (B2g)

Note: The bolded modes were found in the Raman spectra of our material while the non bolded refer to the other Raman modes of P25 which were not observed in our composites (Balachandran, U. and Eror, N., 1982. Raman spectra of titanium dioxide. *Journal of Solid State Chemistry*, 42(3), pp.276–282.).

Photocatalytic Degradation of Diclofenac Sodium and Carbazole (Intermediate Compound) by HCP25

The maximum formation of carbazole dimer was observed at 5 min of experiment. This value was used as a maximum and degradation of carbazole during the experiment is calculated based on that absorbance value.









Emission Spectra of the UV-A, UV-B and Visible Lamps Used in the Experiments

Figure S11. Emission spectra of UV-A, UV-B and visible lamps used. Integrated area values represent the value of total irradiance of the lamps.