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## Supporting Information

# Hydrothermal synthesis of highly porous carbon monoliths from carbohydrates and phloroglucinol

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#### FTIR spectroscopy: Experimental details

Fourier Transform Infrared (FTIR; Shimadzu, IRAffinity-1) spectroscopy measurements were performed on KBr pellets containing about 1 wt.% of aerogel, at the following operating conditions: 10 scans; resolution =  $4 \text{ cm}^{-1}$ .

#### **FTIR spectroscopy: Discussion**

Figure S1 shows FTIR spectra recorded for organic aerogels obtained using a phloroglucinol/monosaccharide ratio of 1/2 after a hydrothermal treatment at 180 °C during 20 h from fructose (HTC-F2\_180), from glucose (HTC-G2\_180), and from xylose (HTC-

X2\_180). These spectra are rather similar, depicting main absorption bands centred at 3430-3440 cm<sup>-1</sup> (O-H stretching), 1700 cm<sup>-1</sup> (C=O stretching), 1630 cm<sup>-1</sup> (-C=C- stretching of aromatic and furanic rings), 1430-1440 cm<sup>-1</sup> (C-C stretching of aromatic rings) and 1020-1030 cm<sup>-1</sup> (symmetric C–O–C stretching of furanic rings). The broad signal between 1200 and 1300 cm<sup>-1</sup> can be attributed to either the C-O(-H) stretching of phenolic compounds, or the asymmetric C-O-C stretching of furanic rings.



**Figure S 1.** FTIR spectra recorded for (a) HTC-F2\_180; (b) HTC-G2\_180; and (c) HTC-X2\_180.

FTIR spectra recorded for organic aerogels obtained from fructose using a phloroglucinol/monosaccharide ratio of 1/7 after a hydrothermal treatment at 180 °C (Figure S2b; HTC-F7\_180) and using a phloroglucinol/monosaccharide ratio of 1/2 after a

hydrothermal treatment at 130 °C (Figure S2c; HTC-F2\_130) do not show any noticeable difference with the ones described previously.



**Figure S 2.** FTIR spectra recorded for (a) HTC-F2\_180; (b) HTC-F7\_180; and (c) HTC-F2\_130.

Figure S3 shows FTIR spectra recorded for the organic aerogel obtained from glucose using a phloroglucinol/monosaccharide ratio of 1/2 after a hydrothermal treatment at 180 °C during 20 h (HTC-G2\_180) and the corresponding carbon aerogel after further thermal treatment at 950 °C under nitrogen (950-G2\_180). The carbon aerogel depicts two main absorption bands centred at 1575 and 1510 cm<sup>-1</sup> associated with the C=C- stretching of aromatic rings. The presence of residual oxygen functional groups is mainly shown by the asymmetric C-O-C stretching band centred at 1250 cm<sup>-1</sup>.



Figure S 3. FTIR spectra recorded for (a) HTC-G2\_180 ; and (b) 950-G2\_180.

#### CO<sub>2</sub> sorption: Experimental details

CO<sub>2</sub> sorption analysis were performed using a QUADRASORB SI/MP and an Autosorb-1 MP (Quantachrome Instruments) at 273 K. Prior to measurement, the samples were degassed at 150 °C for 20 h. Grand Canonical Monte Carlo (GCMC) method was used. The data evaluation was done using the QuadraWin 5.05 and Origin 8.0 software. High purity gases were used for all measurements.

	CO <sub>2</sub> sorption (GCMC model)	
Carbogels	$S_{GCMC}$ (m <sup>2</sup> .g <sup>-1</sup> )	$V_{p} (cm^{3}.g^{-1})$
950-G2_180	935	0.27
950-X2_180	1064	0.35

Table S 1. CO<sub>2</sub> sorption data, calculated *via* the GCMC method, assuming carbon slit pores.



**Figure S 4.** CO<sub>2</sub> sorption isotherms (273 K) and pore size distribution (calculated using CO<sub>2</sub> adsorption data *via* the GCMC method, assuming carbon slit pores) of thermally treated carbogels (a-b) 950-X2\_180 and (c-d) 950-G2\_180.

### X-ray diffraction: Experimental details

X-ray diffraction (XRD) patterns were recorded with RINT Ultima III apparatus (Rigaku Corp., Japan) operating with  $CuK_{\alpha 1}$  radiation ( $\lambda$ = 1.54 Å). Samples were ground and measured on deepened plastic samples holders.



Figure S 5. XRD patterns of (a) 950-G2\_180 and (b) 950-X2\_180 carbon aerogels.

#### Raman spectroscopy: Experimental details

Raman spectra were recorded at room temperature and acquired using a Jobin-Yvon T-6400 spectrometer (Horiba, Ltd., Japan) under a confocal microscope Olympus Bx41. The 532 nm emission line of an argon laser was used for excitation. Samples were ground prior to measurements.



Figure S 6. Raman spectra of (a) 950-G2\_180 and (b) 950-X2\_180 carbon aerogels.

#### **Raman spectroscopy: Discussion**

The partial graphitization of the carbon aerogels was confirmed by Raman spectroscopy. The two bands centered at approximately 1345 and 1590 cm<sup>-1</sup> can be attributed to defect sp<sup>3</sup> carbons (D band) and graphite-like sp<sup>2</sup> carbons (G band), respectively.