Supplementary information

Sustainable synthesis of nanoporous carbons from agricultural waste and their application for solid-phase microextraction of chlorinated organic pollutants

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E-mail: yrbian@issas.ac.cn (Yongrong Bian), jiangxin@issas.ac.cn (Xin Jiang). Address: NO. 71 East Beijing Road, Nanjing, 210008, PR China. Fig. S1 Synthesis scheme of the NPCs.



The hydrothermal process could resolve OSRS into small fragments, forming oxygen-contained functional groups and increasing the porosity by hydrolysis of the lignin and hemicellulose. Then, remained crystalline cellulose was activated by KHCO₃ under the process of redox reactions between various decomposed potassium compounds with carbon precursor, blowing the melt carbon matrix with as-prepared H_2O and CO_2 , intercalating into the carbon lattices of the carbon matrix with the formation of metallic K, and removing metallic K via acid washing.

Fig. S2 Typical SEM (a, b) and TEM (c, d) images of the NPC-8.



Fig. S3 Typical TG and DSC results for the mixture of the hydrochar and KHCO₃ before and after activated.





Fig. S4 XRD patterns of the NPCs.



Fig. S5 Raman spectrum of the NPCs.



Fig. S6 FT-IR spectrum of the NPCs.

Fig. S7 Typical XPS spectra (a), C 1s XPS spectra (b) and N 1s XPS spectra (c) from the NPC-4.



Fig. S8 Effects of operation conditions on the extraction efficiencies of the NPC-coated fiber (NPC-8) towards CBs and PCBs, desorption temperature (1a, 1b), extraction temperature (2a, 2b) and extraction time (3a, 3b).



Uncertainty	Calculation	Parameters	
Standard preparation		Δ mi: Error associated to the measurement of a	
	$\cdots = \sum (A \min / \min) 2 1 1 / 2$	given parameter	
	μ_{sp} [$(\Delta m/m)$]	mi: Value measured in each of those actions	
		$SD_{x/y}$: Residual standard deviation in the	
		determination of the sample	
Calibration curve		n: Number of the measurements carried out for a	
	$\mu_{cal} = (SD_{x/y}/p) \{ (1/n) + (1/p) + [(y-y_a)^2/b^2/(p-1)/S_{xi}^2] \}^{1/2}$	given sample	
		p: Number of the points included in the	
		calibration curve	
		y _a : Average value of the analytical signal	
		b. Stope of the canoration curve	
		S_{xi} : Variance of the standards concentration	
Precision	$\mu_{pre}=SD/n^{1/2}$	SD: Standard deviation between duplicate	
		samples	
		n: Number of the replications	
Accuracy		RSD: Relative standard deviation of the average	
	$\dots - \mathbf{P} \mathbf{S} \mathbf{D} / \mathbf{s}^{1/2}$	percent recovery	
	$\mu_{ac} = \kappa_{SD} / \pi^{-2}$	n: Number of the replications	
Expanded uncertainty	$U_{(k=2)} = 2C(\mu_{sp}^{2} + \mu_{cal}^{2} + \mu_{pre}^{2} + \mu_{ac}^{2})^{1/2}$	C: Average concentration of the analyte	

Table S1 Description of main contributions and respective calculation formula forthe expanded uncertainty (U) following bottom-up approach.

	Limits of detections, ng L^{-1}							
Analytes	This study	LLE-DSPE-	SPNE-	SBSE-	SPME-	HS-SPME-	SPME-	HS-SPME-
		GC-ECD ¹	GC-ECD ²	GC-MS ³	GC-MS ⁴	GC-ECD ⁵	GC-ECD ⁶	GC-MS ⁷
1,3,5-TCB	0.34		_	_	_	2.25	_	_
1,2,3-TCB	0.19	—	—	—	—	0.94	—	—
1,2,3,4-TeCB	0.09	—	—	—	—	0.32	—	—
1,2,3,5-TeCB	0.28	—	—	—	—	—	—	—
PeCB	0.64		—	—	—	0.50	—	—
НСВ	0.30	_	—	—	0.69	0.64	—	_
PCB-8	0.29	_		0.36		—		
PCB-9	0.22	—	—	—	—	—	—	—
PCB-18	0.21	—	—	—	—	—	—	0.03
PCB-20	0.08	—	—	0.39	—	—	—	—
PCB-28	0.13	0.25	1.40	0.50	—	—	0.1	—
PCB-52	0.12	0.30	3.10	0.27	—	—	0.1	0.03

Table S2 Comparison of developed method with other analysis methods fordetermination of selected CBs and PCBs in water samples.

Table S3 Comparison of partly detected COPs from water samples with others in the literatures.

Detected concentration (ng L^{-1})						
Analytes	This study	Rainwater ⁸	lake water1 4	pond water 9	lake water2 10	well water 11
1,3,5-TCB	0.45	_	_	_	nd	0.6
HCB	5.88	11.7	17.5	14.8	nd	0.3

nd: not detected; rainwater: collected from in Guangzhou, China, on May 21st, 2014; lake water1: collected from the surface water of Pearl River, Guangzhou, China; pond water: collected from the surface water of pond located in Sun Yat-sen University, China; lake water2: collected from the surface water of Xuan Wu Lake, Nanjing, Jiangsu, China; well water: none.

Table S4 Advantages and drawbacks of diverse protocols for the determination ofCOPs from water.

Techniques	Advantages	Drawbacks		
LLE-DSPE-GC-ECD ¹	High sensitivity	Complex procedure		
	Short extraction time consuming: 10 min	Uses organic solvents: methanol, n-hexane, dichloromethane		
	Multiple analytes at the same run: 7	Consumes large quantities of solvents: 100 mL		
	Low cost of analysis instrument	Large sample volume: 1000 mL		
SPNE-GC-ECD ²	Short extraction time consuming: 9.5 min	Complex procedure		
	Consumes small quantities of solvents: 0.1 mL	Moderate sensitivity		
	Multiple analytes at the same run: 7	Uses organic solvents: n-hexane		
	Low cost of analysis instrument			
	Small sample volume: 1 mL			
SBSE-GC-MS ³	Easy and simple procedure	Long extraction time consuming: >24 h		
	High sensitivity	High cost of analysis instrument		
	Non-toxic solvents	large sample volume: 200 mL		
	Multiple analytes at the same run: 77			
	Environmental friendly, benign			
HS-SPME-GC-ECD	Easy and simple procedure	Long extraction time consuming: 24.5 min		
	High sensitivity			
	Non-toxic solvents			
	Multiple analytes at the same run: 12			
	Environmental friendly, benign			
	Low cost of analysis instrument			
	Small sample volume: 10 mL			

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