Beihsner, J., **Schierz, A.**, **Balda, M.**, **Georgi, A.** (2025):
Development and evaluation of a novel activated carbon material for use as an in-situ sorbent for PFAS-contaminated sites
Vom Wasser **123** (3), 83 - 84

https://doi.org/10.1002/vomw.202500012

Development and evaluation of a novel activated carbon material for use as an *In-situ* sorbent for PFAS-contaminated sites

J. Beihsner, Leipzig/Germany, A. Schierz, Leipzig/Germany, M. Balda, Leipzig/Germany, A. Georgi, Leipzig/Germany

Helmholtz Centre for Environmental Research, Department of Technical Biogeochemistry, Permoser Str. 15, 04318 Leipzig/Germany

1. Introduction

Per- and polyfluoroalkyl substances (PFAS) are known to be extremely difficult to remove from the environment, due to their exceptional persistence and (high) mobility in water. The *In-situ* application of (colloidal) activated carbon (C(AC)) is designed to act as a barrier, preventing the further migration of PFAS within aquifers. This approach demands high-performance activated carbon materials capable of significantly slowing PFAS movement, even at low sediment loading levels (0.1–1 wt.%), to ensure long-term effectiveness lasting decades.

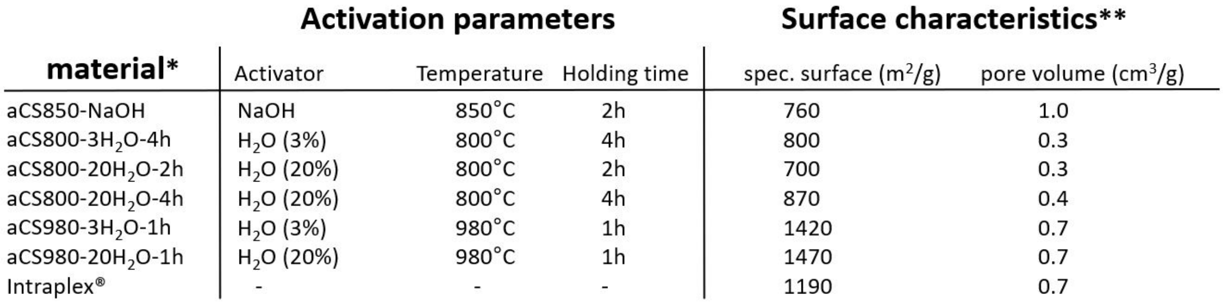
This study focuses on the development and evaluation of injectable CAC for use in *In-situ* sorption barriers. Two materials are investigated: a research-stage activated carbon, Activated Carbon Spheres (ACS) and the commercially available CAC Intraplex®. ACS are synthesized through a bottom-up process using sucrose, a renewable and locally accessible raw material, via hydrothermal carbonization followed by thermal activation at low water vapor partial pressure [1]. This method produces high-purity spherical carbon particles with an average diameter of approximately 1 µm and a microporous structure. By adjusting the synthesis and pyrolysis parameters, the pore size and surface chemistry of ACS can be tailored to meet specific application needs. In contrast, commercial CAC is typically manufactured through a top-down approach, involving the wet grinding of granular or powdered activated carbon. AC is often derived from non-renewable raw materials such as lignite or hard coal.

This study focuses on optimizing ACS material for the adsorption of perfluorooctanoic acid (PFOA), a key representative of PFAS. Previous findings indicate that both pore structure and surface properties - specifically a low oxygen content - are critical for achieving high PFOA adsorption performance. However, de-functionalized surfaces are susceptible to aging when exposed to aqueous environments. To address this, the aging behaviour of ACS produced under various conditions and its impact on sorption properties were analyzed. Future research will also examine how other water constituents (e.g. organic matter, salts, co-contaminations) influence PFAS adsorption.

2. Experimental part

ACS were synthesized using a bottom-up approach, where sucrose underwent hydrothermal carbonization followed by activation through chemical agents (e.g., H_3PO_4, NaOH) or physical activation with water vapor at varying temperatures and holding times (Table 1 [1]). The resulting ACS materials were then analyzed using various analysis methods (e.g. nitrogen physisorption, temperature-programmed decomposition (TPD), Raman spectroscopy) to determine their physical-chemical properties.

Table 1: Materials tested and their surface characteristics (*aCS temperature-activation agent-holding time, ** N_2-adsorption).



	Activation parameters			**Surface characteristics****	
material*	Activator	Temperature	Holding time	spec. surface (m^2/g)	pore volume (cm^3/g)
aCS850-NaOH	NaOH	850°C	2h	760	1.0
aCS800-3H_2O-4h	H_2O (3%)	800°C	4h	800	0.3
aCS800-20H_2O-2h	H_2O (20%)	800°C	2h	700	0.3
aCS800-20H_2O-4h	H_2O (20%)	800°C	4h	870	0.4
aCS980-3H_2O-1h	H_2O (3%)	980°C	1h	1420	0.7
aCS980-20H_2O-1h	H_2O (20%)	980°C	1h	1470	0.7
Intraplex®	-	-	-	1190	0.7

Sorption isotherms for PFOA were investigated through batch experiments under various environmentally relevant conditions. For comparison, the commercial activated carbon CAC Intraplex® (Intrapore GmbH) was included. PFOA concentrations were analyzed using liquid chromatography coupled with mass spectrometry (LC-MS).

Based on their sorption performance, two ACS materials were selected for an ageing study. These materials were exposed to three different storage conditions: nitrogen atmosphere, air and anoxic tap water. The sorption behavior of the pristine and aged carbon materials was then compared.

3. Results and discussion

Sorption isotherms for PFOA on various ACS were modeled using the Freundlich equation. From these isotherms, single-point sorption coefficients (K_d) for PFOA were calculated at equilibrium concentrations of 50 µg/L (as a concentration typical for contaminated sites) and 1 mg/L (approaching the maximum loading range of the carbon) (Figure 1 left). The sorption performance of chemically and physically activated ACS materials (with the exception of aCS800-20H_2O-2h) was comparable to that of commercial CAC. Additionally, the adsorption of six perfluoroalkyl acids (PFAAs) and perfluorosulfonic acids (PFSAs) was examined (Figure 1 right). Under the test conditions, chemically activated ACS (aCS500-NaOH) removed 40% of perfluorobutanoic acid (PFBA) at a carbon dosage of 1.2 g/L, while physically activated ACS materials achieved removal rates exceeding 65%, in most cases even >95%. The best ACS material shows a sorption coefficient for PFBA as short-chain PFAS of $10^{4.7}$ L/kg (c_e = 7.9 µg/L) which is excellent.

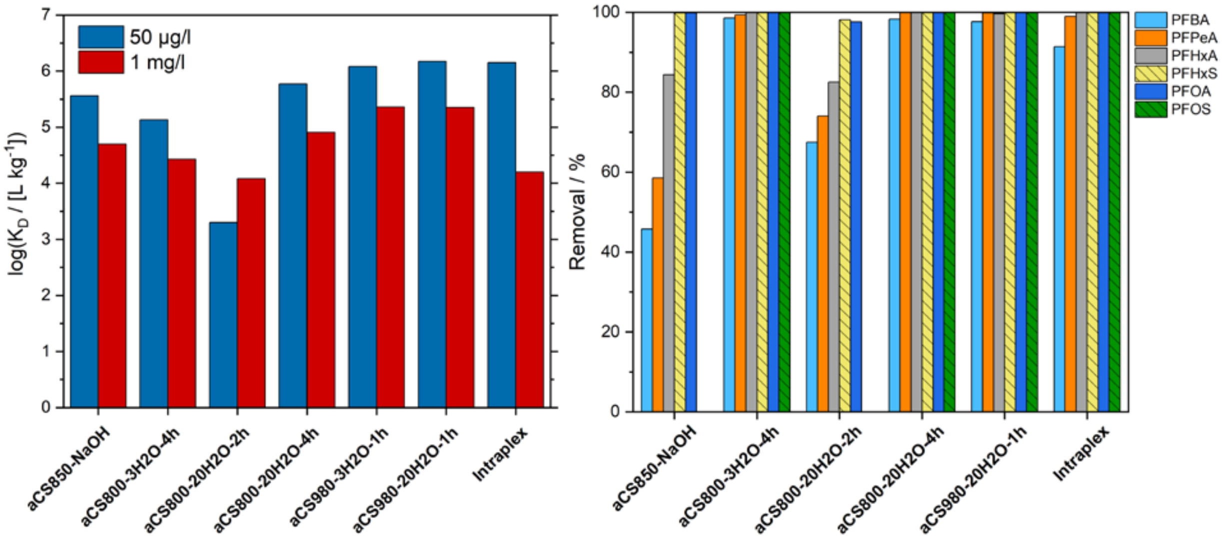


Figure 1: (left) Single-point partition coefficient for PFOA at $c_{e,PFOA}$ = 50 µg/l and 1 mg/l, pH = 6.5 10 mM Na_2SO_4 and (right) removal of 6 PFAS from 10 mM Na_2SO_4 at pH 6.5 (c_{ACS} = 1.2 g/L and c_{total} = 600 µg/L per compound resulting in aqueous phase equilibrium concentrations of c_e = < 0.02 to 325 µg/L).

The high activation temperature was identified as the most critical factor for high sorption affinity and capacity for PFAS. This can be partly explained by the increase in specific surface area and pore volume (Table 1), but is also related to a higher degree of graphitization and defunctionalisation of the activated carbon material. Previous studies have shown that a highly defunctionalised surface of the activated carbon is advantageous for the adsorption of PFAS anions [2]. This is due to the positively charged surface sites created by proton adsorption on condensed aromatic regions with highly delocalized pi-electrons.

Since ACS particles are intended for environmental applications in groundwater remediation, maintaining the stability of their beneficial surface and pore structures over time is critical. The ageing of ACS has been studied over a short timeframe, corresponding to low oxidation rates. Initial findings after 2 months of ageing indicate differences in PFOA sorption properties. These experiments are ongoing, and updated results, along with detailed characterization of both fresh and aged materials, will be presented. Analysis of the oxygen content in the aged materials is currently in progress.

HTC synthesis followed by pyrolysis enables the production of tailor-made ACS from renewable raw materials. By optimising the activation process, the sorption properties of ACS can be tailored specifically for PFAS sorption. In addition, the goal of improving the ageing resistance of the material - and thus its long-term usability - was successfully achieved. The results shown will guide activated carbon design for optimal performance in the adsorption of PFAS.

References

[1] Balda, M., Mackenzie, K., Woszidlo, S., Ulig, H., Möllmer, J., Kopinke, F.-D., Georgi, A., 2023: Bottom-up synthesis of de-functionalized and dispersible activated carbon spheres as colloidal adsorbent, Int. J. Mol. Sci., 24 (4), art. 3831

[2] Saeidi, N., Kopinke, F.-D., Georgi, A, 2020: Understanding the effect of carbon surface chemistry on adsorption of perfluorinated alkyl substances, Chem. Eng. J., 381, 122689

Acknowledgements: The work is founded by Promotionsstipendium of the Deutsche Bundesstiftung Umwelt.