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Targeted testing of activated carbons for advanced wastewater treatment

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HIGHLIGHTS

- Novel test method for selection and quality control of powdered activated carbon.
- Inexpensive and fast photometric quantification of target pollutants in test solution.
- Selectivity of activated carbon for one of two groups of organic micro-pollutants.
- Good correlation of micro-pollutant removal in the test solution and in treated wastewater.

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ABSTRACT

Powdered activated carbon (PAC) is a promising agent for the adsorptive removal of organic micro-pollutants (OMP) from wastewater treatment plant (WWTP) effluents. Reliable and predictive test methods are required for the selection and for the quality control of PAC. In the present investigation, a pragmatic test method using indicative target OMP and photometric analyses was developed. Eight different PAC were compared according to the test method and according to removals of individual OMP in WWTP effluent. The test method allowed identification of the PAC with the highest and the lowest removals of benzotriazole and elucidated differences of the PAC with regard to removals of individual OMP. In WWTP effluent both positive and negative correlations between OMP removals with different PAC were observed: Some PAC removed diclofenac, carbamazepine and metoprolol to a comparatively greater extent while other PAC are comparably more efficient for benzotriazole, methylbenzotriazole and acesulfame adsorption.

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1. Introduction

The application and consumption of numerous organic substances such as pharmaceuticals result in the occurrence of organic micro-pollutants (OMP) in wastewater and, after wastewater treatment, in the receiving aquatic environment. Biological processes during wastewater treatment are not sufficient for the complete removal of recalcitrant OMP [1–3]. Although the ecological risks emanating from OMP are not fully elucidated yet, additional wastewater treatment steps for the preventive OMP removal are being considered. One of the most promising options in both wastewater and drinking water treatment is the adsorptive removal of OMP by powdered activated carbon (PAC) [4–8].

PAC is typically derived from different raw materials such as coal, lignite, coconut shell and others. The large inner surface of PAC results from activation by thermal treatment. The raw materials, the processing and the activation vary and thus the quality of activated carbons might differ from one batch to the other. In order to assure a continuous high quality, practical but reliable testing methods for PAC are required.

The specific surface area (B.E.T. surface) is a typical characteristic for the description of PAC although its reliability has been questioned [9]. However, the surface area does not provide information about the pore size and pore size distribution but the latter have been reported to significantly influence the adsorption efficiency [10–11]. Several standardized testing methods have been developed to classify PAC or to predict the applicability of a PAC:

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phenole, tannin, molasses or methylene blue numbers [12–13], iodine number [14–15] or nitrobenzene number [15]. However, several of these conventional characterizations have been found not to be predictive for the removal of relevant OMP from treated wastewater [16].

The absorption of visible or ultraviolet light by organic compounds due to the excitation by light energy provides valuable information about dissolved organic carbon (DOC). Color reactions of inorganic ions and specific reagents with subsequent photometrical quantification are widely applied. Compared to analytical systems consisting of liquid chromatography coupled with mass spectrometry, a photometer is non-expensive and available in most wastewater treatment plants. The UV absorption is a very robust parameter but the sensitive and respective quantification limits of UV spectrometers are comparably high, much higher than trace concentrations. Advanced UV and visible light spectrophotometers can be used for the quantification of individual compounds in mixtures by applying complex data processing, so called derivative spectrophotometry [17–19]. However, if the absorption spectra of individual OMP are sufficiently different, a conventional photometer enables the quantification of the respective concentration in a solution containing a limited number of OMP in high and thus measurable concentrations.

This investigation aimed at developing a simple but meaningful test procedure for the selection or rejection of adsorbents that only requires basic equipment and thus can be widely applied without great expenses (compare Fig. S1 in the Supplementary data). Acesulfame (ACS), benzotriazole (BTA) and diclofenac (DCF) were chosen for the test since they are very persistent and proposed as indicators substances for adsorption processes [20–21]. The concentrations of these target compounds in test solutions were adjusted to levels that can be quantified by UV spectrometry (without expensive mass spectrometry). Eight different activated carbons were tested and the results of the test procedure were compared to results obtained in WWTP effluent.

2. Materials and methods

2.1. Powdered activated carbon

Eight different PAC of different source materials including black coal, charcoal, lignite, coconut shell and peat have been tested. The commercial products and some characteristics provided by the manufacturers are listed in Table 1. Stock suspensions (1 g/L) of all PAC were prepared in ultra-pure water.

2.2. Batch tests

Single solute test solutions of 2 mg/L acesulfame potassium ($C_4H_4KNO_4S$, 201.2 g/mol), 2 mg/L benzotriazole ($C_6H_5N_3$, 119.1 g/mol) or 2 mg/L diclofenac ($C_{14}H_{11}Cl_2NO_2$, 296.2 g/mol) and a solution containing all three compounds (2 mg/L each) were prepared with ultra-pure water. The comparatively high concentrations of OMP were required for their photometric quantification. No further chemicals were added. For comparison batch tests were conducted in effluent from a Berlin WWTP which contained 6.5 μg/L BTA, 4.2 μg/L ACS, 3.2 μg/L formylaminoantipyrine (FAA), 2.8 μg/L DCF, 2.7 μg/L methylbenzotriazole (MBT), 2.4 μg/L metoprolol (MTP), 1.2 μg/L carbamazepine (CBZ) and 0.3 μg/L sulfamethoxazole (SMX). No additional OMP were spiked to the effluent. Further information and structural formulas for all considered OMP are provided in Table S1 in Fig. S2.

Batch tests were conducted in glass flasks containing 50 mL test solution. After addition of 1 mL (corresponding with 20 mg/L) of the PAC stock suspensions with a micropipette, the flasks were intensively agitated on a horizontal shaker. After 30 min contact time the PAC was removed by filtration (0.45 μm pore size). Contact times of 30 min are economically feasible for advanced wastewater treatment [4] and thus appropriate for a test method, although equilibrium is not reached [6,22].

2.3. Analytics

Single and triple solute test solutions were analyzed by a dual beam spectrophotometer (Lambda 12 UV–VIS photometer, Perkin Elmer, Germany) in 1 nm steps in the scanning mode for the range from 220 to 300 nm. Preliminary calibrations confirmed that UV light absorbance was linearly correlated with the concentrations of ACS, BTA and DCF, independent from the wavelength.

OMP in WWTP effluent were quantified by high pressure liquid chromatography (2.5 μm XSelect HSS T3 50 × 2.1 mm column, Waters, USA) coupled with a tandem mass spectrometer (TSQ Vantage, Thermo Fisher, USA). OMP were quantified according to internal deuterized standards for each OMP. UV light absorption at 254 nm (UV_{254}) was measured with the spectrophotometer specified above.

2.4. OMP quantification in mixed solutions

Two different methods were applied to quantify the concentrations of the individual OMP according to photometrical measurements after adsorption tests in mixed solutions: (1) solving a system of linear equations or (2) approximation by the solver function in Microsoft Excel.

2.4.1. System of linear equations

The resulting UV light absorption A at each wavelength λ in a solution containing ACS, BTA and DCF is the sum of the individual absorptions (Eq. (1)):

$$A_{\lambda,MIX} = A_{\lambda,ACS} + A_{\lambda,BTA} + A_{\lambda,DCF} \quad (1)$$

The UV light absorption $A_{\lambda,OMP}$ of a certain OMP at a given wavelength λ can be described with a wavelength specific absorption coefficient $\varepsilon_{\lambda,OMP}$ and the path length l (Eq. (2)):

$$A_{\lambda,OMP} = \varepsilon_{\lambda,OMP} \cdot c_{OMP} \cdot l \quad (2)$$

Table 1
Details of the eight powdered activated carbons tested in the present study.

Abbreviation	Product name	Supplier	Source material	B.E.T. surface (m^2/g)	Ash content (%)
AP	Carbopal AP	Donau Carbon, Germany	Lignite	1300	–
AZ	AZ 1050	CSC, Germany	Black coal	>1050	<10
CCP	Carbopal CCP 90 D	Donau Carbon, Germany	Coconut shell	1000–1100	<5
HK	HK 950	CSC, Germany	Charcoal	>950	<8
HKP	HKP 1050	CSC, Germany	Charcoal	>1050	<10
MB	Carbopal MB 4	Donau Carbon, Germany	Charcoal	900	<9
PF	AquaSorb 5000 PAC-F	Jacobi Carbons, Germany	Lignite	1150	<18
SAE	SAE Super	Norit, Germany	Mixture	1150	–

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