Rapid small-scale column testing of granular activated carbon for organic micro-pollutant removal in treated domestic wastewater

F. Zietzschmann, J. Müller, A. Sperlich, A. S. Ruhl, F. Meinel, J. Altmann and M. Jekel

ABSTRACT

This study investigates the applicability of the rapid small-scale column test (RSSCT) concept for testing of granular activated carbon (GAC) for organic micro-pollutants (OMPs) removal from wastewater treatment plant (WWTP) effluent. The chosen experimental setup was checked using pure water, WWTP effluent, different GAC products, and variable hydrodynamic conditions with different flow velocities and differently sized GAC, as well as different empty bed contact times (EBCTs). The setup results in satisfying reproducibility and robustness. RSSCTs in combination with WWTP effluent are effective when comparing the OMP removal potentials of different GAC products and are a useful tool for the estimation of larger filters. Due to the potentially high competition between OMPs and bulk organics, breakthrough curves are likely to have unfavorable shapes when treating WWTP effluent. This effect can be counteracted by extending the EBCT. With respect to the strong competition observed in GAC treatment of WWTP effluent, the small organic acid and neutral substances are retained longer in the RSSCT filters and are likely to cause the majority of the observed adsorption competition with OMPs.

Key words | adsorption, breakthrough curve, granular activated carbon, organic micro-pollutants, rapid small-scale column test, wastewater

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INTRODUCTION

Organic micro-pollutants (OMPs) are nowadays found in wastewater treatment plant (WWTP) effluents, surface waters and, to some extent, also drinking waters (Heberer 2002; Reemtsma *et al.* 2006). Since there is little knowledge on long-term or combinatory effects of trace-level amounts of OMPs in the different water bodies (Dieter & Mückter 2007), many water services plan additional water treatment steps for preventive OMP removal (Jekel *et al.* 2013). Among these additional steps are mainly oxidizing processes such as ozonation, and adsorptive processes like activated carbon adsorption (Margot *et al.* 2013; Altmann *et al.* 2014). With respect to WWTP effluent treatment, many studies focus on powdered activated carbon (Boehler *et al.* 2012; Zietzschmann *et al.* 2014a), but also granular activated carbon (GAC), which has been applied successfully in drinking and reservoir water treatment (Ridal *et al.* 2001), is discussed as a possible technique (Gimbel *et al.* 2011; Boehler *et al.* 2013).

Testing of powdered activated carbon products is comparatively simple (experiments with stirred or shaken batches containing the respective water and powdered activated carbon), whereas tests with GAC are more complex and therefore require more planning and time, usually involving bench- or pilot-scale filters. Simplification of this setup can be approached by a number of means. Equilibrium batch experiments with pulverized GAC can be used to assess the activated carbon capacity but this approach does not account for the relevant mass transfer processes in GAC filters. Therefore, mass transfer models were developed which simulate the transport of adsorbates in GAC filters. However, this approach requires somewhat numerous input parameters which cannot always be provided, especially when modeling multi-solute problems (Weber & Smith 1987).

Most of these shortcomings can be avoided using rapid small-scale column tests (RSSCTs) which aim at reducing

doi: 10.2166/wst.2014.357

the experiment size, the water consumption, and the time effort that are generally encompassed in pilot/large-scale filter experiments (Crittenden *et al.* 1986; Crittenden *et al.* 1991). RSSCTs are scaled-down from pilot/large filters by using smaller sized GAC particles whilst keeping the hydrodynamic regime in a range that allows for direct comparability of the small and the large filters. RSSCTs account for many of the effects that cannot be accounted for by using equilibrium batch experiments and mass transfer modeling, and thus are a promising technique when needing estimation of GAC filter performance.

MATERIALS AND METHODS

Water samples and pre-treatment

All chemicals and reagents were of reagent grade or better. Ultra-pure water (resistivity >17 MΩ * cm) was produced from fully de-ionized water using an Ultra Maxima (Elga Berkefeld Lab Water, Germany). Effluent was sampled from the WWTP at Ruhleben, Berlin, Germany. In order to reduce the head loss that occurred due to filter blocking with suspended substances, the WWTP effluent was filtered with a SIEMENS Memcor PVDF 200 kDa membrane. The initial concentrations of the OMPs were left as in the WWTP effluent (which is the reason why they vary between some of the results shown).

Activated carbons

The used GAC product was Jacobi Aquasorb 5000 G (and, where indicated, Norit 1240 and CSC HC AZ 1050). To obtain the desired reduced GAC grain sizes, the products were ground and sieved. Equilibrium experiments were conducted with pulverized GAC.

Equilibrium experiments

Equilibrium experiments were conducted using a typical bottle point method (Zietzschmann *et al.* 2014a). Single solute (OMPs spiked into ultra-pure water) and multi-solute (WWTP effluent) data were fitted using the Freundlich isotherm equation. In multi-solute systems this results in pseudo single solute fitting. This approach gives satisfactory fitting results for adsorbates whose isotherm/ loading curves have continuous positive slopes (no bending to negative slopes at high adsorbate concentrations/low activated carbon dosages).

RSSCT setup

Glass columns (inner diameter 8 mm) were used as filters, and glass pearls/glass wool was used to hold the GAC in place. Depending on the dimensioning of the respective RSSCT filter, the bed volumes were 0.5 mL (1 cm bed height) and 1 mL (2 cm bed height). (With these bed volumes, throughputs of, for example, 100,000 bed volumes can be obtained with as little as 50 and 100 L of water, respectively.) To dimension the RSSCT filters, the constant diffusivity approach was used (Crittenden *et al.* 1986; Crittenden *et al.* 1991). The main equations necessary for the downscaling are given in Equations (1) and (2). The empty bed contact time (EBCT) and the filter velocity/hydraulic loading (v) of the small column (SC) and the large column (LC) are related through the ratios of the corresponding particle diameters (d_P). Since the GAC particle sizes of the SC can cause high head losses, the Reynolds number of the SC can be changed from that of the LC (Crittenden *et al.* 1991), as long as it is kept above 1 (referred to as $Re_{SC,min}$, cf. Equation (2)).

$$EBCT_{SC} = EBCT_{LC} \times \left(\frac{d_{P,SC}}{d_{P,LC}}\right)^2 \quad (1)$$

$$v_{SC} = v_{LC} \times \frac{d_{P,LC}}{d_{P,SC}} \times \frac{Re_{SC,min}}{Re_{LC}} \quad (2)$$

Note that the RSSCT results shown in the current study stem from *seemingly* short EBCTs. Using Equation (1), the EBCTs of equivalent large-scale columns can be inferred, to obtain a more practical impression. Table 1 gives an exemplary overview on corresponding EBCTs on small-scale and large-scale columns for a particle diameter ratio of $d_{P,SC} \approx d_{P,LC}/10$, which results in an EBCT ratio of 1/100.

Breakthrough modeling and calculations

The software FAST 2.0 (Sperlich *et al.* 2008) was used to model GAC filter breakthrough in a pseudo single solute approach.

Carbon usage rates (CURs) were calculated according to Equation (3) where ρ_b is the apparent bed density and $BV_{10\%}$

Table 1 | Exemplary EBCTs for equivalent small-scale and large-scale columns, according to Equation (1); $d_{P,SC} \approx d_{P,LC}/10$

$EBCT_{SC}$	[s]	2	5	10
$EBCT_{LC}$	[s], [min]	200, 3.3	500, 8.3	1000, 16.7

is the number of bed volumes treated until a modeled breakthrough of 10% of the inlet concentration.

$$\mathrm{CUR} = \frac{\rho_b}{\mathrm{BV}_{10\%}} \tag{3}$$

Analytics

Organic micro-pollutants

The OMP concentrations were measured using high performance liquid chromatography with tandem mass spectrometry (HPLC-MS/MS) using an XSelect HSS T3 column (2.5 µm, 2.1 * 50 mm, Waters, USA) with a linear gradient (0.5 mL/min, ultra-pure water with 0.1% HCOOH and 5% methanol versus methanol) and a TSQ Vantage (Thermo Scientific, USA) using electrospray ionization. Two mass fragments were chosen for each analyte (Wasserchemische Gesellschaft 2013), and deuterated isotopes (TRC, Canada; Dr. Ehrenstorfer, Germany) were used for quantification.

Ultraviolet absorption at 254 nm and fractionized dissolved organic carbon

UV absorption was measured on a Lambda 12 (Perkin-Elmer, USA). Fractionized dissolved organic carbon (DOC) was determined using liquid size-exclusion chromatography on an HW50S column (Toyopearl, Japan) with online carbon detection (LC-OCD; DOC-Labor Huber, Germany). The DOC fractions were allocated according to the literature (Huber *et al.* 2011). Data were evaluated using the software ChromCalc (DOC-Labor Huber, Germany).

RESULTS AND DISCUSSION

The single solute (ultra-pure water) and multi-solute (WWTP effluent) Freundlich parameters of selected OMPs are summarized in Table 2. From the data shown for the single solute systems, the order of the adsorbate affinity towards activated carbon is benzotriazole > diclofenac > carbamazepine. The multi-solute Freundlich coefficients are almost two orders of magnitude smaller than the single solute parameters. Also, carbamazepine and diclofenac, which have different single solute parameters, have similar multi-solute parameters. Benzotriazole has increased parameters compared to diclofenac in the single solute system but reduced parameters in the multi-solute system. The results indicate that the multi-solute system, which includes competition between the OMPs and the bulk organics, is too complex to be predictable from the single solute data (Table 2).

The relative UV absorption and relative OMP concentrations are plotted against the throughput of four RSSCTs filled with two GAC products (each in duplicate) in Figure 1. The breakthrough curves shown in Figure 1 demonstrate that there is no full retention of either of the shown parameters. The UV absorption reaches 90% of the initial concentration earlier than 5000 bed volumes of throughput. Among the OMPs, benzotriazole has the strongest retention (50% of the initial concentration at ~20,000 bed volumes) while carbamazepine and diclofenac both reach 50% of the initial concentration earlier than 10,000 bed volumes. The duplicate RSSCTs of the respective GAC product yield nearly identical breakthrough curves. These results show that the chosen experimental setup is reproducible and yields a satisfying comparability.

The observed GAC filter breakthrough behaviors shown in Figure 1 cannot be adequately predicted solely by using equilibrium data. The single-solute Freundlich parameters suggest a stronger retention for diclofenac than for carbamazepine whereas the RSSCT shows a different order of OMP adsorption. The multi-solute Freundlich parameters suggest that carbamazepine and diclofenac should have similar breakthrough behavior. These discrepancies between the information from equilibrium batch experiments and the information from RSSCTs underline the high relevance of mass transfer processes in GAC filters, which need to be considered in adequate breakthrough curve determination. Also, the potential for GAC pore fouling cannot be determined from batch experiments with pulverized GAC (Corwin & Summers 2010).

Table 2 | Single solute and multi-solute (pseudo single solute) Freundlich parameters for selected OMPs; K_F in $(\mu g/mg)*(L/\mu g)^{(1/n)}$

	Benzotriazole		Carbamazepine		Diclofenac	
Solute system	**Single**	**Multi**	**Single**	**Multi**	**Single**	**Multi**
Freundlich coefficient (K_F)	251	2.3	159	3	251	3
Freundlich exponent ($1/n$)	0.45	0.27	0.31	0.3	0.36	0.28

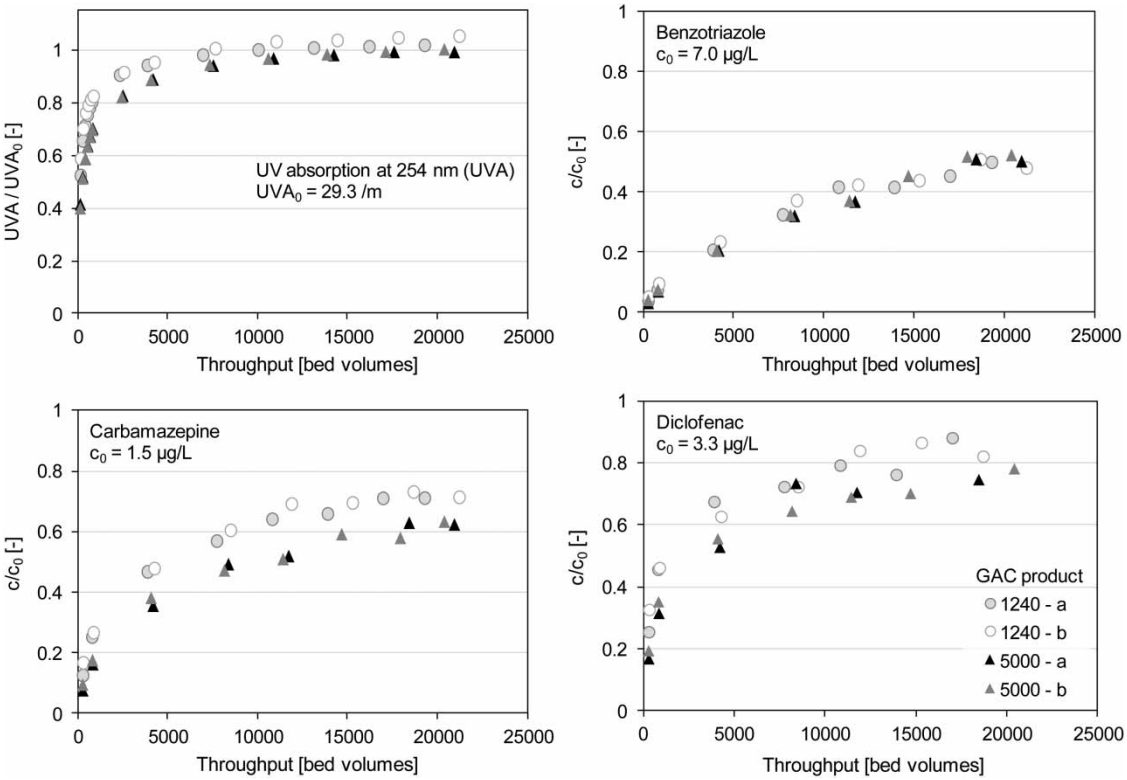

Figure 1 | UV absorption and benzotriazole, carbamazepine, and diclofenac concentrations (relative to the respective inlet WWTP effluent values) versus throughput, with two different GAC products in duplicate (Norit 1240 and Aquasorb 5000), EBCT: 2.2 s, d_p: 125–200 µm.

With respect to the robustness of RSSCTs, the influences of the external and internal mass transfer must always be considered within the framework of the respective setup. Although the external mass transfer can be regulated by the filter velocity, the internal mass transfer cannot be influenced. Thus, high external mass transfer rates are generally desirable. Firstly, they result in good transport of adsorbates to adsorbent particles. Secondly, when the external mass transfer rate is high compared to the internal mass transfer rate, the breakthrough curve is only dependent on the internal mass transfer, resulting in robust experimental setups. In that case, variations of the filter velocity have no consequences on the breakthrough curve appearance. The relative filter effluent concentrations of benzotriazole and diclofenac, adsorbing from WWTP effluent, are plotted against the throughputs of two RSSCTs equipped with the same GAC product at two different bed lengths and different filter velocities, resulting in the same EBCTs, in Figure 2. Both of the RSSCTs yield similar breakthrough curves which are not affected by the differences in the hydrodynamic regimes. Accordingly, the external mass transfer is of minor importance for the RSSCTs in the chosen configuration. These results show that using an approach with a reduced Reynolds number is applicable. Reducing the Reynolds number in order to minimize the head loss, the bed length, and the needed water volume does not affect the RSSCT results within the chosen setup.

A major aspect of the RSSCT concept is to transfer results produced with filters containing small-sized GAC (i.e. RSSCTs) to filters with larger sized GAC, for performance prediction. To check the transferability, the RSSCT equations (Equations (1) and (2)) can be used to relate GAC filters (containing differently sized GAC particles) to each other and evaluate the similarity of their breakthrough curves. The concentrations, relative to the influent WWTP effluent concentration, of benzotriazole and diclofenac at the filter outlets of two RSSCTs with differently sized GAC particles and different EBCTs (related via Equations (1) and (2)) are depicted versus the throughput in Figure 3. The breakthrough curves of the two differently dimensioned RSSCTs show a high similarity. Accordingly, the downscaling from a larger GAC filter to a smaller GAC filter appears valid. The chosen setup can only demonstrate the feasibility of the downscaling to some extent since the particle diameter of the smaller GAC fraction is only ~0.5 times the size of the particle diameter of the large GAC

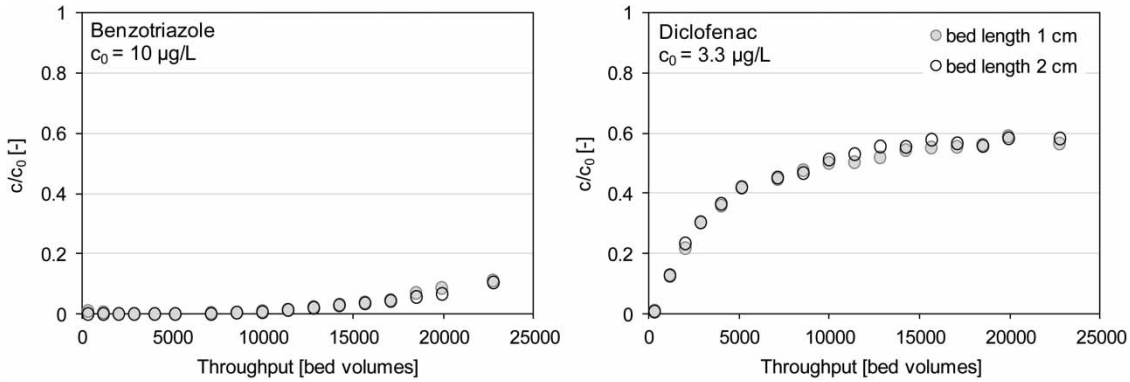

Figure 2 | Benzotriazole and diclofenac concentrations, relative to the respective inlet WWTP effluent values, versus throughput of two RSSCTs with different bed length and different filter velocities (v_1 ~6 m/h, v_2 ~12 m/h) and same EBCT (6 s), GAC Norit 1240, d_P: 90–140 µm.

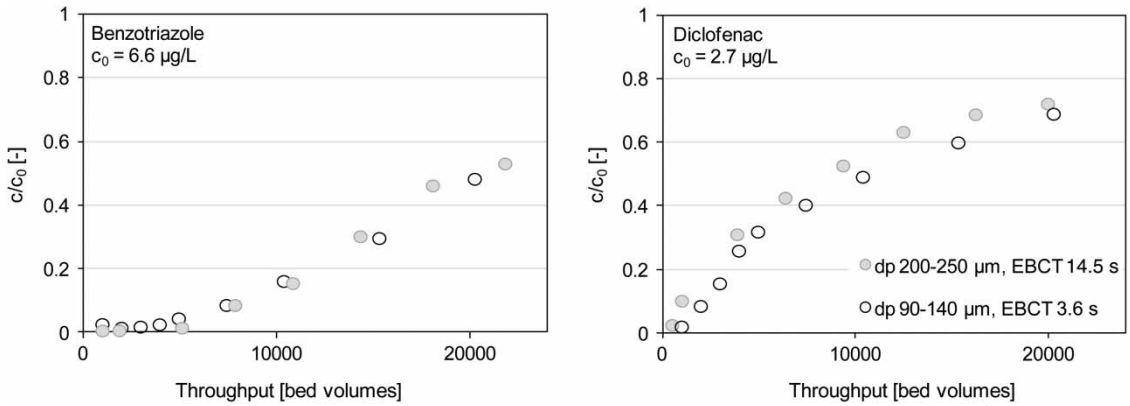

Figure 3 | Benzotriazole and diclofenac concentrations, relative to the respective inlet WWTP effluent values, versus throughput in two equivalent RSSCTs with differently sized GAC and different EBCTs.

fraction. Additional RSSCTs using GAC fractions with larger differences in their particle sizes are necessary to broaden the observed feasibility of the downscaling. However, the high similarity of the determined breakthrough curves underlines that small-scale filters which are scaled down from large filters using the RSSCT concept can produce results that allow for rough prediction of large-scale filters. Also, these results show that the chosen constant diffusivity approach (diffusivity of adsorbates is independent of the GAC particle size) appears feasible here (Crittenden *et al.* 1986).

The previous results showed adequate reproducibility and robustness, and the feasibility of reducing the SC Reynolds number in order to minimize experimental efforts. Furthermore, scaling between GAC filters/RSSCTs with differently sized GAC particles is valid within the experimental conditions used here. However, most of the breakthrough curves obtained from treating WWTP effluent diverge from the typically preferred S-shaped form (Figures 1–3). A comparison of these WWTP effluent breakthrough curves with breakthrough curves from the treatment of spiked ultra-pure water (cf. Supporting information, available online at http://www.iwaponline.com/wst/070/357.pdf) confirms that the appearance of the WWTP effluent breakthrough curves does not originate from design errors or short-circuiting of the RSSCTs, but the reason is adsorption competition between the OMPs and the bulk organics contained in the WWTP effluent. The DOC concentration in the Berlin Ruhleben WWTP effluent is usually 10–12 mg/L, thus about 10^3–10^5 times the concentration of most OMPs, which explains the potentially high competition. The early OMP breakthrough and flat shapes of the breakthrough curves are highly unfavorable because they result in high CURs (mass of carbon used per volume of water treated) and low efficiency if specific OMP removals are targeted (e.g. c/c_0 must be <0.1 at any time). It is therefore highly relevant to delay the OMP breakthrough (and to minimize the CURs) and maximize the GAC filter efficiency.

The relative concentrations of benzotriazole and diclofenac from RSSCTs with different EBCTs are plotted against the throughput in Figure 4; note that the scaling of the abscissae is relative and does not depict absolute volumes but the numbers of bed volumes. The graphs demonstrate that an increase of the water–GAC contact time yields longer retention of the OMPs and an improved shape of the breakthrough curves. This causes a reduction of the CUR (less GAC mass per volume of water/more water treated per mass of GAC). Accordingly, the EBCT is a crucial parameter when dimensioning GAC filters for WWTP effluent treatment, and longer EBCTs cause improved retention of adsorbates and higher efficiency. Therefore, when treating WWTP with potentially strongly competing bulk organics, the EBCT should possibly be maximized to obtain efficient CURs. However, maximizing the EBCT means increasing filter beds in any application where a specific volumetric flow shall be treated, and accordingly the financial and technical efforts increase simultaneously. A balance between GAC filter efficiency and expenses must thus be found for any particular application/problem.

Using the RSSCT results, scale-up modeling using the software *FAST 2.0* is possible. Figure 4, right, shows the CURs at a breakthrough of 10% for benzotriazole and diclofenac in relation to the EBCT. For both OMPs, the CURs decline with increasing EBCT, especially in the EBCT range below 30 min. Accordingly, the maximization of the EBCT in constellations with high adsorption competition such as the treatment of WWTP effluent can improve CURs. Thus, reducing the volumetric flow or increasing the bed length in GAC treatment of WWTP effluent should be considered where possible. Pilot experiments with different WWTP effluents and EBCTs within the range of the EBCTs modeled in the current study showed CURs in a similar range as reported here (Gimbel *et al.* 2011; Boehler *et al.* 2012). However, a direct comparison is not possible because of the strongly varying setups of the different studies (GAC products, WWTP effluents, backwashing).

The aforementioned strong competition between OMPs and bulk organics is unlikely to be caused equally by all of the bulk organics because the compounds comprising the bulk organics have different molecular composition, resulting in varying size, chemistry etc. (Zietzschmann *et al.* 2014b). Thus, different fractions of the bulk organics (the latter usually being encompassed in surrogate parameters such as DOC) cause different competition and accordingly have different affinities onto activated carbon. The relative concentrations of the bulk organic fractions as identified by LC-OCD are depicted versus the throughput of an exemplary RSSCT in Figure 5, left, together with the chromatograms of the corresponding blind WWTP effluent sample and the RSSCT outlet after 10 min/200 bed volumes (Figure 5, right). The biopolymers and humics fractions show nearly no retention and reach relative concentrations (c/c_0) of 0.9 at <200 bed volumes and ~1,200 bed volumes, respectively. The smaller fractions of the acids and neutrals demonstrate stronger retention and reach $c/c_0 \approx 0.9$ at >5000 and 3500–4000 bed volumes, respectively. The retention of the bulk DOC (bypass-DOC) lies in-between the retention of the weakly and strongly adsorbable fractions. The breakthrough curves in Figure 5 show that the small organic substances of the acid and neutral fractions have a comparatively strong affinity towards the GAC in the RSSCT filter. Accordingly, these substances are presumably causing the major competitive effects in the adsorption of OMPs from WWTP effluent, which confirms other studies (Zietzschmann *et al.* 2014b).

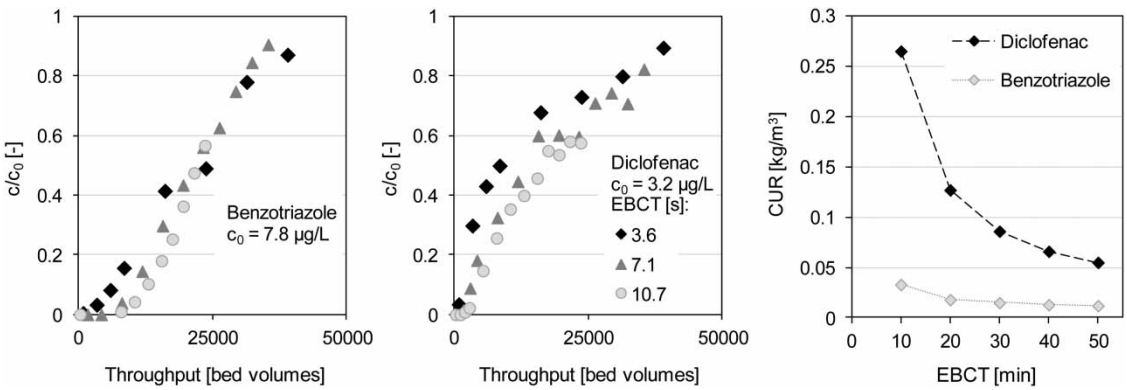

Figure 4 | Left and middle: benzotriazole and diclofenac concentrations, relative to the inlet WWTP effluent values, versus throughput at varying EBCT, d_P: 90–140 µm; right: modeled CURs at 10% breakthrough for benzotriazole and diclofenac in relation to the EBCT.

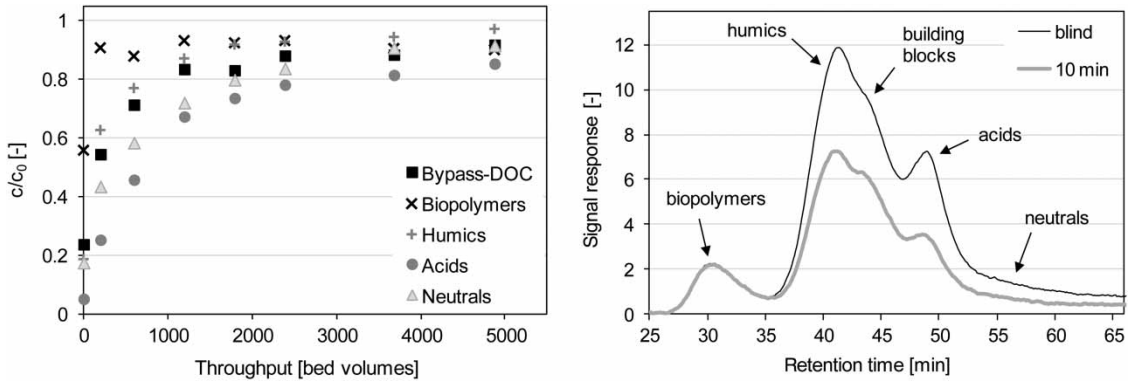

Figure 5 | Left: DOC ('bypass-DOC') and DOC fraction concentrations, relative to the inlet WWTP effluent values, versus throughput; GAC AZ 1050, d_p: 125–200 μm, EBCT (3 s); right: LC-OCD chromatograms of blind WWTP effluent sample and RSSCT outlet after 10 min, with fraction denominators (Huber *et al*. 2011).

CONCLUSION

The RSSCT concept for GAC testing in OMP removal from WWTP effluent was shown to be applicable. The reproducibility and robustness are satisfying within the chosen experimental setup. RSSCTs can be used to compare different performances of GAC products in OMP removal from WWTP effluents. The performances of larger filters in WWTP effluent treatment can be estimated using RSSCTs. Due to potentially high concentrations of bulk organics in WWTP effluents, the competition for OMP adsorption can be substantial. This competition causes unfavorable OMP breakthrough curves (flat shape, early OMP appearance in the filter outlet) and high CURs. Extending the EBCT can improve the shape of the breakthrough curves and therefore can reduce CURs and increase the efficiency of GAC filters. It was shown that small organic acid and neutral substances are retained longer on the RSSCT filters than larger DOC compounds, and we conclude that these substances are responsible for the majority of the observed adsorption competition with OMPs. Future experiments should use GAC particles with larger differences in the large and small GAC filters in order to further verify the transferability of RSSCT results to large-scale filters in WWTP effluent treatment.

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First received 1 July 2014; accepted in revised form 31 July 2014. Available online 23 August 2014