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Removal of micropollutants and nutrients in household wastewater using organic and inorganic sorbents

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ABSTRACT

The efficiency of five organic and five inorganic sorbents in removing 19 organic micropollutants (MPs), phosphorus, nitrogen, and dissolved organic carbon (DOC) was tested in a two-week column experiment using household wastewater spiked with pharmaceuticals (n = 6), biocides/pesticides (n = 4), organophosphates (n = 3), a fragrance, a UV-stablizer, a food additive,a rubber additive, a plasticizer and a surfactant. Two types of granular activated carbon (GAC), two types of lignite, a pine bark product, and five mineral-based sorbents were tested. All the organic sorbents except pine bark achieved better removal efficiencies of DOC (on average, 70 ± 27%) and MPs (93 ± 11%) than the inorganic materials (DOC: 44 ± 7% and MPs: 66 ± 38%). However, the organic sorbents (i.e. GAC and xyloid lignite) removed less phosphorus (46 ± 18%), while sorbents with a high calcium or iron content (i.e. Polonite® and lignite) generally removed phosphorus more efficiently (93 ± 3%). Ammonium-nitrogen was well removed by sorbents with a pH between 7 and 9, with an average removal of 87%, whereas lignite (pH 4) showed the lowest removal efficiency (50%). Some MPs were well removed by all sorbents (≥97%) including biocides (hexachlorobenzene, triclosan and terbutryn), organophosphates (tributylphosphate, tris-(1,3-dichloro-2-propyl)phosphate and triphenylphosphate) and one fragrance (galaxolide). The pesticide 2,6-dichlorobenzamide and the pharmaceutical diclofenac were poorly removed by the pine bark and inorganic sorbents (on average, 4%), while organic sorbents achieved high removal of these chemicals (87%).

Keywords: Micropollutants (MPs); Synthetic substances; Sorbents; On-site sewage facilities (OSSFs)

1. Introduction

Organic micropollutants (MPs) comprise a vast number of man-made and natural substances, such as pharmaceuticals, personal care products, pesticides, and industrial chemicals, which pose a threat for the aquatic environment over the world [1]. Besides, many MPs are not completely removed during wastewater treatment due to their physicochemical properties [1]. Many studies have been performed on the removal of MPs in centralized waste water treatment plants (WWTPs) worldwide [2–4]. However, less attention has been given to on-site sewage facilities (OSSFs), even though OSSFs are commonly used in decentralized rural and semi-urban areas. The concentrations of MPs in OSSF

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effluents were generally comparable to those observed in conventional WWTP effluents [6,7]. In Sweden, 753 000 households (corresponding to 10% of the population) are using OSSFs and are not connected to municipal WWTPs [5]. These OSSFs are commonly soil based systems, such as soil and infiltration beds [5], where sand is currently the most prevalent filter medium. Sand was capable of removing some MPs, while a number of other MPs were poorly removed [8,9]. A variety of wastewater treatment technologies have been studied for the removal of MPs, including membrane bioreactors [10–12], activated sludge systems [10], UV oxidation [13], ozonation [14], slow sand filtration [8], and sorbents such as activated carbon (AC) [15]. However, for practical and economic reasons, most of these technologies are not suitable for OSSFs. Other concerns include ecotoxicological hazards when using reactive treatments, e.g. ozonation may generate toxic transformation products that increase the genotoxic and mutagenic potential of MPs in wastewater [16,17]. Thus, there is an urgent need to study alternative filter materials with better adsorption capacities than sand or add-on filter materials that can be used after soil based systems to remove MPs from OSSF discharge.

The discharge of nutrients from the OSSFs to receiving water bodies is an important environmental issue. In Sweden, the OSSFs release 295 tons of phosphorus and 3066 tons nitrogen per year [18]. Furthermore, two studies showed that the discharged water from several OSSFs cannot meet the protection levels recommended by the Swedish Environmental Protection Agency [18,19]. Therefore, studying the removal of nutrients in OSSFs treatments is an important issue.

AC is one of the most effective organic sorbents for removing MPs. This material has been tested for, e.g., pharmaceuticals [15,20,21], cholorophenoxy pesticides [22] and per- and polyfluoroalkyl substances [23,24]. The granular activated carbon form (GAC) is more suitable for OSSFs than the powdered form (PAC), since it can be used as a filter medium and also because it requires less mechanical treatment. However, the adsorption capacity can differ between different types of GAC [15,25]. Moreover, GAC is expensive, so an alternative sorbent with lower costs is desirable. Other sorbents, such as lignite (often referred to as brown coal), has been studied for its adsorption of different substances, including MPs, and good removal efficiencies have been observed [26–29]. However, the performance of these organic sorbents in the removal of nutrients has not been explored yet. Furthermore, even though most inorganic sorbents are applied for nutrients removal, only few studies reported their efficiency in the removal of MPs. Different clay materials showed promising adsorption potential for the removal of a few pharmaceuticals and personal care products from aqueous solutions [30–32], and Zeolite, which has a porous uniform structure, showed good removal of some MPs, such as methyl tert-butyl ether (MTBE) [27,33]. However, the performance of these materials has only been confined to a limited number of individual MPs. Sand is the most common filter medium used in OSSFs. Nevertheless, improvements have to be made to enhance both nutrients and MPs removal. Thus, further research including a broader range of MPs as well as nutrients by a variety of sorbents is needed to improve wastewater treatment in OSSFs.

In order to identify proper sorbents for OSSFs for the removal of both MPs and nutrients, ten different sorbents were tested in a column experiment, covering materials with different physicochemical properties and application purposes. Based on literature studies, GAC and lignite showed promising removal for several types of MPs, therefore GAC with different particle sizes, lignite with different physical characteristics (coal and fiber) and a natural wood fiber product were used in the experiment. The studied inorganic sorbents are commercial products used for phosphorus/nitrogen/organic matters removal in OSSFs. Sand was chosen as a reference material to represent typical soil bed systems for OSSFs.

The main aim of this study was to find alternative sorbents for OSSFs which can remove both MPs and nutrients. A short-term column experiment was performed to evaluate the selected sorbents in terms of their capacity for removing multiple MPs, as well as nutrients (dissolved organic carbon (DOC), total phosphorus (P_{tot}) and ammonium nitrogen (NH_4-N)) and to provide an overview of their merits and demerits. The MPs tested in the experiment covered a wide range of chemicals, with different physicochemical properties, including pharmaceuticals, biocides/pesticides, fragrance, UV-stabilizer, food additives, rubber additives, plasticizers, surfactants and organophosphates.

2. Materials and methods

2.1. Filter media

The selected filters comprised five organic and five inorganic materials, including natural materials and industrially processed materials (Table 1). The selection was based on literature studies, practical applications, and economical benefits [15,20–33].

The five organic filter materials comprised two kinds of GAC, two kinds of lignite, and a natural pine bark product (Zugol®). The materials Filtrasorb®300 and Envirocarb™ 207EA, lignite and Xylitare coal-based sorbents, whereas Zugol® is a natural wood fiber. Both GACs (i.e. Filtrasorb® 300 and Envirocarb™ 207EA) were manufactured from bituminous coal, but have different particle sizes (0.6–2.4 and 3–4 mm, respectively). In order to achieve a raw compact lignite sorbent, this material was crushed and sieved to 2–4 mm and used as filter material. Xylit consists of natural wood fibers derived from lignite (usually called xyloid lignite), and Zugol® is made of Swedish pine bark without the addition of any chemicals.

Rådasand is a natural sand excavated from the Råda esker (south-west Sweden), and was washed and sieved to 0.7–1.0 mm (referred to as sand in the following). Filtralite® P and Polonite® are used in OSSFs to remove phosphorus, while Filtra® N is intended to remove nitrogen. Unlike the other inorganic filter materials, Sorbulite® and Filtra® N are porous materials, therefore providing a large adsorptive surface area and increasing the possibility for removal of MPs.

2.2. Target compounds

The target compounds included 19 MPs, covering the following chemical classes: biocides/pesticides (n = 4), a

Table 1
Filter materials used in the column experiment along with supplier, particle size, surface area, pore volume and average pore size

Filter media	Material	Supplier	Particle size[a] (mm)	Surface area[b] ($m^2 g^{-1}$)	Pore volume[b] ($cm^3 g^{-1}$)	Average pore size[b] (nm)
Organic materials						
Filtrasorb® 300	GAC: agglomerated bituminous coal	Chemviron Carbon AB, Sweden	0.6–2.4	783.5	0.519	2.7
Envirocarb™ 207EA	GAC: bituminous coal	Chemviron Carbon AB, Sweden	3–4	914.4	0.507	2.2
Lignite	Brown coal	MátraiErömü, Bükkábrány, Hungary	2–4	5.3	0.020	14.7
Xylit	Nature wood fibers derived from lignite	Eloy Water, Belgium	Fibers	2.5	0.010	16.7
Zugol®	Swedish pine bark	Zugol AB, Sweden	Fibers	2.5	0.017	26.4
Inorganic materials						
Rådasand	Sand: Quartz and feldspar	Rådasand AB, Sweden	0.7–1.0	0.6	0.002	17.0
Sorbulite®	Tobermorite (autoclaved aerated concrete)	Ecofiltration Nordic AB, Sweden	2–4	20.4	0.092	18.1
Filtra® N	Zeolite (clinoptilolite and mordenite)	Nordkalk AB, Sweden	1–4	19.0	0.067	14.1
Polonite®	Calcium silicate bedrock	Ecofiltration Nordic AB, Sweden	2–6	3.8	0.022	23.1
Filtralite® P	Expanded clay aggregate	Saint-Gobain Byggevarer AS, Norway	0.5–4	0.5	0.003	24.2

[a]Provided by supplier; [b]The specific surface area, pore volume and average pore size of the sorbents was determined by Brunauer-Emmett-Teller (BET) analysis using a Tristar surface area analyzer.

Table 2
Chemicals (n = 19) spiked to the feed water with abbreviation and class name used for the column experiments

Analyte	Abbreviation	Class
Hexachlorobenzene	HCB	Biocides/pesticides
Triclosan	TCS	
2,6-Dichlorobenzamide	BAM	
Terbutryn	TBT	
α-Tocopheryl acetate	α-TPA	Food additive
Galaxolide	HHCB	Fragrance
Tributylphosphate	TBP	Organophosphates
Tris-(1,3-dichloro-2-propyl)phosphate	TDCPP	
Triphenylphosphate	TPP	
Carbamazepine	CBZ	Pharmaceuticals
Oxazepam	OZP	
Metoprolol	MTP	
Diclofenac	DF	
Losartan	LST	
Caffeine	CF	
N-Butylbenzenesulfonamide	n-BBSA	Plasticizer
2-(Methylthio)benzothiazole	MTBT	Rubber additive
2,4,7,9-Tetramethyl-5-decyn-4,7-diol	TMDD	Surfactant
Octocrylene	OC	UV-stabilizer

food additive, a fragrance, organophosphorus compounds (n = 3, used as e.g. flame retardants), pharmaceuticals (n = 6), a plasticizer, a rubber additive, a surfactant and a UV-stabilizers (Table 2). The MPs were selected based on their environmental significance and occurrence in OSSFs discharges based on previous studies [6,7].

2.3. Experimental set-up

The feed water for the column experiment was taken from the effluent of a soil bed system serving 13 households located at Drottningholm close to Stockholm, Sweden. The facility was constructed in 2012 and consists of a three-chamber septic tank followed by a soil bed. Two standard mixtures were added into the feed water. Standard Mixture 1 contained HCB, TCS, α-TPA, HHCB, TBP, TDCPP, TPP, n-BBSA, MTBT, TMDD and OC. Standard Mixture 2 contained BAM, TBT, CBZ, OZP, MTP, DF, LST and CF. To prepare the feed solution, 3 mL of Standard Mixture 1 and 5 mL of Standard Mixture 2 were added to the wastewater in a 2-L volumetric flask and mixed thoroughly with a magnetic stirrer. The mixture was then added to 8 liters of wastewater in a 10 L flask which resulted in a spiking concentration of 0.55 µg L^{-1} to 35 µg L^{-1} for individual MPs (for details see Table S2). The concentrations of the selected MPs were measured after spiking as well (Table S3). Feed solutions were prepared freshly in the beginning of each week and the experiment lasted for two weeks.

The columns used (n = 11) consisted of PP tubes with internal diameter of 4.82 cm (Fig. 1). Each column was filled with a 10 cm layer of one of the test filter media. The reference column was kept empty. Two multichannel pumps were used to apply the feed water with a vertical saturated flow and a pumping rate adjusted to 1.14 mL min^{-1} for each column (Fig. 1). To simulate realistic wastewater flows, the pumps were run three times per day, from 7:00 to 7:30 h, 12:00 to 13:00 h, and 18:00 to 18:30 h. The surface load was 75 L $m^{-2}d^{-1}$. Unspiked wastewater was pumped into one empty reference column to determine background levels of the MPs. Feed water was pumped onto the top of each column. Effluent pipes were curved to form a 'U' shape and raised 10 cm above the column base. This shape ensured that the filter media were saturated during the experiment. Effluent water from each column was collected separately in 250 mL glass bottles, transferred daily to sample glass bottles for respective weekly samples, and stored in the refrigerator at 4°C. At the end of the experiment, the concentration of 19 MPs were analyzed in 26 samples including 2 unspiked influent samples, 2 spiked influent samples, 20 effluent samples from filter columns and 2 effluent samples from the reference column.

2.4. Analytical methodology

HCB, TCS, α-TPA, HHCB, TBP, TDCPP, TPP, n-BBSA, MTBT, TMDD and OC were extracted and analyzed according to Blum et al. [6]. Briefly, the wastewater samples were filtered, extracted by automated solid phase extraction with OASIS HLB cartridges (200 mg, 6 mL, Waters, Milford, MA, USA) and filtered through Na_2SO_4 columns before gas chromatography mass spectrometry analysis (Pegasus 4D HRT, Leco Corp., St.Joseph, MI, USA). BAM, TBT, CBZ, OZP, MTP, DF, LST and CF were analyzed by off-line SPE, using Oasis HLB (500 mg, 6 mL, Waters Corporation, Milford, MA, USA) cartridges, followed by Ultra-High-Performance-Liquid Chromatography (Acquity UHPLC, Waters Corporation, Milford, MA, USA) coupled to quadrupole-time-of-flight mass spectrometry (QTOF Xevo G2S, Waters Corporation, Manchester, UK). Extracts were analyzed in both positive and negative electrospray ionization mode. Details of the analytical method can be found in Gros et al. [7]. Quantification was carried out with the isotope dilution method using a mixture of labelled internal standards (Table S1).

The water quality parameters analyzed included DOC, ammonium-nitrogen (NH_4-N), phosphate-phosphorus (PO_4-P), total phosphorus (P_{tot}), pH, turbidity, and conductivity. Analysis of DOC was carried out with a TOC-L TOC analyzer (Shimadzu, Kyoto, Japan) and of NH_4-N, PO_4-P and P_{tot} were analysed using Seal Analytical AA3 Autoanalyzer.

2.5. Calculations and statistical analysis

The removal efficiency (RE) of water quality parameters was calculated according to:

$$RE = \left(1 - \frac{C_{eff}}{C_{in}}\right) \times 100\% \quad (1)$$

where C_{in} is the influent concentration of the water quality parameter, and C_{eff} is the effluent concentration of the water quality parameter.

Release/adsorption of MPs from/onto the sorbents was assessed by calculating the MP removal efficiency (RE_{MPs}). The removal efficiencies were corrected for potential levels of the MPs in the system according to:

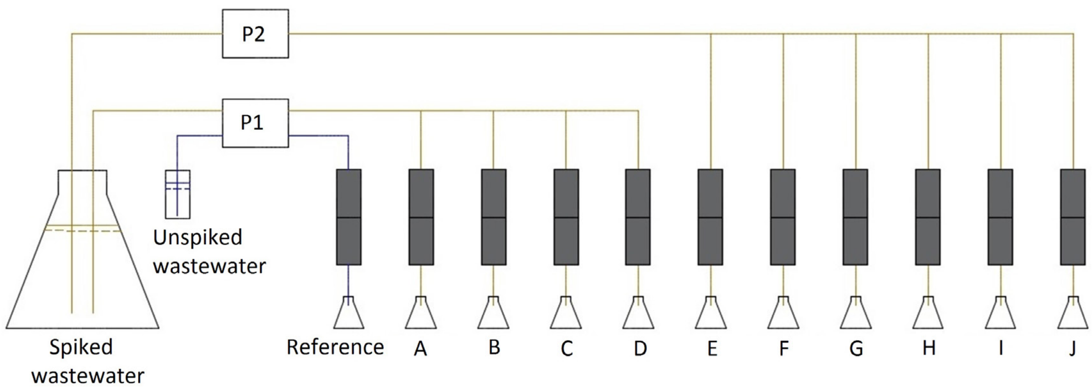

Fig. 1. Schematic of the column experiment including five organic and five inorganic sorbents (note: not to scale). Name of the sorbents (from left to right): (A) Filtrasorb®300, (B) sand, (C) Xylit, (D) Filtra®N, (E) lignite, (F) Filtralite®P, (G) Zugol®, (H) Sorbulite®, (I) Envirocarb™ 207EA, (J) Polonite®.

$$C_{ch} = C_{in} - C_{eff0} \quad (2)$$

where C_{ch} is the changed concentration of the MP in the outflow of the reference column, C_{in} is the influent concentration of the MP, and C_{eff0} is the effluent concentration of the MP in the reference column. RE_{MPs} was calculated according to:

$$RE_{MPs} = \left(1 - \frac{C_{eff}}{C_{insp} - C_{ch}}\right) \times 100\% \quad (3)$$

where C_{eff} is the concentration of the MP in the outflow from respective column C_{insp} is the concentration of the MP in spiked influent wastewater, and C_{ch} is the changed concentration of the MP in the outflow of the reference column.

Experimental results were statistically evaluated using SPSS (IBM). Principal component analysis (PCA) was performed to evaluate the variation in removal behavior of the studied chemicals by the ten sorbents. Cronbach's alpha was calculated to test the reliability of the extracted components. One-way ANOVA and Least Significant Difference (LSD) post-hoc test were performed to test whether the removal efficiency differed between sorbents and chemicals. Negative removals were considered as zero removals. The removal efficiency was considered as the dependent variable, while spiked MPs and sorbents were the independent variables. The relation between MP removal and pore size and surface area of the sorbents was tested using Spearman's rank correlation.

2.6. QA/QC

The equipment was run for one week with unspiked wastewater before the experiment started in order to condition the filters and test the function of the set-up. The equipment contained some plastic and silicon materials, e.g., in pumping tubes, which could not be avoided. Since the manufacturing process of these materials was unknown, there was a risk that they contained chemicals that could have contaminated the effluent water samples. The impact of this on the experimental set-up was checked by measuring the compounds in the influent and effluent water of the reference column.

A large amount of n-BBSA was released into the outflow from the pumps and experimental columns, therefore the removal of n-BBSA was not considered in the analysis and evaluation of filter materials. The release and adsorption of other chemicals were minor compared with the spiked concentration (≤5%).

For Standard Mixture 1 compounds, method validation results for the GC-MS analysis including recovery experiments, linearity, and precision can be found in Blum et al. [6]. Laboratory blanks were extracted in parallel to the samples. In general, the blank levels were below the limit-of-quantification (LOQ) except for TMDD (37 ng L^{-1}) and αTPA (19 ng L^{-1}). For Standard Mixture 2 compounds, method performance parameters for the compounds analyzed by UHPLC-QTOF included recovery efficiencies, linearity, method precision, method detection (MDLs) and quantification limits (MQLs) as well. These parameters can be found in Gros et al. [7]. Compounds quantification was performed by using linear regression calibration curves and the internal standard approach, to account for possible matrix effects. Internal standards used for each compound are indicated in Table S1. Calibration standards were measured at the beginning and at the end of each sequence, and one calibration standard was measured repeatedly throughout the sequence, after every 20–25 samples to check for signal stability. Method blanks were performed to account for any background levels of the analytes investigated, and they consisted of Milli-Q water, and these blanks were analyzed following the same extraction procedure as real samples.

3. Results and discussion

3.1. Water quality parameters

After spiking with the case chemicals, the DOC concentration of the original feed increased from 10 mg L^{-1} to 440 mg L^{-1}, which means the 98% of the DOC came from the solvent of the mixtures of MPs. The organic sorbents GAC Filtrasorb®300 and GAC Envirocarb™207EA achieved the best removal of DOC among all sorbents (Fig. 2A), with an average removal efficiency of 97% and 95%, respectively. Among the other organic filter materials, lignite and Xylit showed intermediate performances, with average DOC removal of 32% and 52%, respectively, while Zugol® removed only 3.0%. This low removal may be due to the pine bark release constituent carbon into the water. Thus, based on the DOC results, GACs had the best potential to remove MPs, whereas Zugol® had the worst removal potential. The effluent concentrations of DOC were quite similar for all inorganic sorbents, which may indicate that they also remove MPs to a similar degree.

The feed water from soil bed effluent contained low levels of NH_4-N (3.8 mg L^{-1} on average) and ranged from 0.3 mg L^{-1} to 1.3 mg L^{-1} in the column effluent (Fig. 2B). The removal of NH_4-N in the sorbents was likely caused by ion exchange or biological nitrification. Zeolite is well known to remove ammonium from wastewater by ion exchange [34]. Filtra® N that consists of zeolite, achieved 90% removal of NH_4-N, thus performed best among all sorbents. Biological nitrification can be impacted by several factors, for instance temperature, pH,and dissolved oxygen level [35]. The temperature during the experiment was around 15°C, which is optimal for nitrification and the optimal pH for nitrification is between 7.5 and 8 [35]. The pH of the lignite was 4, while Polonite® and Filtralite®P had pH values > 10, which can inhibit the nitrification [35]. The impact of pH was reflected in NH_4-N removal, as lignite, Polonite®, and Filtralite®P had the lowest removal efficiency (50%, 71%, and 74%, respectively). Other sorbents had pH values between 7 and 9 and achieved an average removal rate of around 87% ± 2%. The oxygen content in the feed water was about 6 mg L^{-1}, which provided sufficient oxygen for nitrification.

The inorganic sorbents were more effective in removing phosphorus than the organic sorbents (Fig. 2C). Sorbents with a high content of calcium, such as Sorbulite® (19% Ca) and Polonite® (25%) [36] achieved good phosphorus removal rates (above 95%), as they were able to provide sufficient Ca^{2+} and OH^- for the formation of calcium phosphate ($Ca-PO_4$) precipitates [37]. Among the organic sorbents, Zugol® and lignite removed a large proportion of phosphorus, e.g., the P_{tot} removal rate was 94% and 89%, respec-

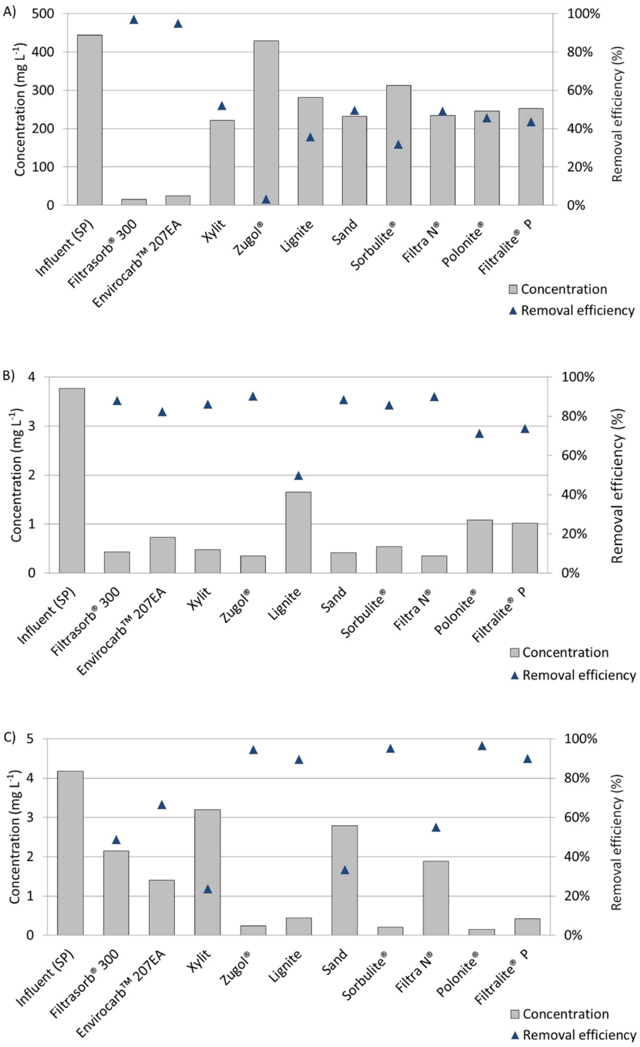

Fig. 2. Mean concentration of three wastewater quality parameters for two weeks: (A) dissolved organic carbon (DOC); (B) ammonium-nitrogen (NH_4-N); (C) total phosphorus (P_{tot}) in the spiked influent and in effluent from the 10 sorbents during two weeks of the experiment. The removal efficiency (RE) at the end of the experiment is shown as blue triangles.

tively. Zugol® contains 20% calcium and lignite contains 14% iron [38], which is beneficial for phosphorus precipitation. The GAC sorbents, Xylit, Filtra® N, and sand were not able to remove phosphorus, so removal when using these materials was probably only due to biological processes. Filtrasorb®300, Envirocarb™207EA and Xylit achieved only 49%, 66%, and 22% reduction in P_{tot}, respectively. Sand, the most commonly used filter medium in soil based system, achieved only 33% removal of P_{tot}.

3.2. Removal of micropollutants (MPs)

Removal efficiencies varied considerably between MPs depending on the sorbent (Fig. 3). Coal-based organic sorbents Filtrasorb®300, Envirocarb™207EA, lignite and Xylit achieved the best removal, with average overall removal

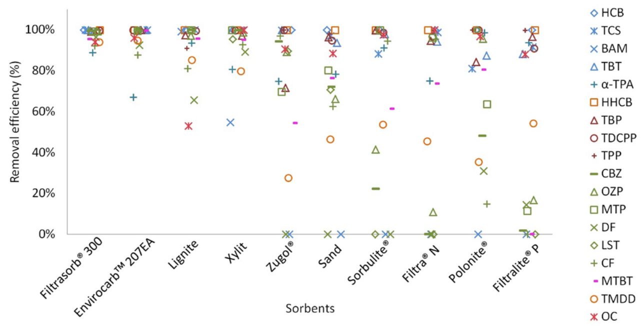

Fig. 3. Average removal efficiency (%) of individual MPs by the 10 sorbents.

efficiencies between 90% and 97%. Natural wood fiber (Zugol®) was less efficient (on average 74%), while the inorganic sorbents were even less efficient in reducing MP levels, with average overall removal efficiencies ranging from 53% to 73%. Sorbent type and chemical characteristics significantly influenced the removal efficiency ($p < 0.05$; one-way ANOVA, Table S3 and Table S4 in the Supporting information).

The individual removal of the MPs by GAC ranged from 88% and 100%, except for α-TPA (78%). The average removal efficiency was 97% for Filtrasorb®300 and 95% for Envirocarb™ 207EA. Lignite and Xylit achieved good removal for most MPs, with average removal efficiencies of 92% and 93%, respectively, except for DF, BAM and OC, which were moderately well removed (Fig. 3), the average removal of the three compounds by Xylit and lignite was 81% and 73%, respectively. Inorganic sorbents showed good removal of several MPs; e.g., both HCB and HHCB were 100% removed by all inorganic sorbents and the average removal of TCS, TBP, TDCPP, TPP and TBT was 95%. However, the other MPs were poorly to moderately removed by inorganic sorbents. For instance, the average removal of CBZ, CF,DF, LST, MTP and OZP was 29%, 53%, 9%, 34%, 70% and 46%, respectively. Filtralite®P showed significantly ($p < 0.05$) lower removal efficiencies (average 53%) than sand, Polonite and all organic sorbents (Table S3). In total 8 out of the 18 tested MPs were poorly removed by Filtralite®P, i.e. less than 20% (Fig. 3).

DF and BAM were significantly different from that of the rest of the MPs ($p < 0.05$; Table S4) with low removal efficiencies by Zugol® and all tested inorganic sorbents, with average removal efficiencies of 8% (DF) and 0% (BAM), respectively. α-TPA, MEP, OC, HCB, TCS, HHCB, TBP, TDCPP, TPP and TBT were significantly different from the rest of the chemicals ($p < 0.05$; Table S4) because of high overall removal efficiencies by coal-based sorbents.

Principal component analysis was carried out to explore the variation in MP removal efficiencies between the different sorbents. The two first principal components (PC1 and PC2) explained 50% and 24% of the variation, respectively (Fig. 4). The Cronbach's alpha value was found to be 0.85 for PC1 and 0.77 for PC2, indicating that the accuracy was acceptable.

In the score plot, the sorbents were clearly divided into two groups. Group 1 contained the organic sorbents GAC Envirocarb™207EA, GAC Filtrasorb®300, and lignite, and

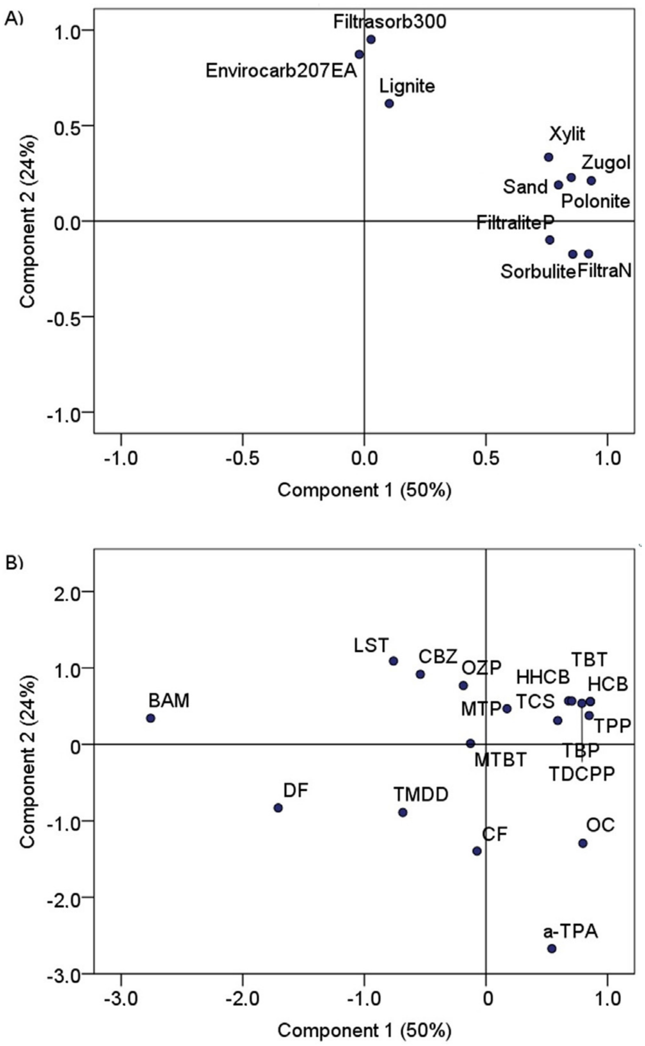

Fig. 4. (A) Scoreplot and (B) loading plot of the MP removal efficiencies using the five organic and five inorganic sorbents.

Group 2 contained all five inorganic sorbents, in addition to the organic sorbents Xylit and Zugol®, demonstrating different removal behavior for the different sorbents.

The chemicals in the upper right corner of Fig. 4B include the biocides (HCB and TCS), organophosphate (TBP, TDCPP, and TPP), a fragrance (HHCB), and a pesticide (TBT). These compounds were well removed by all sorbents, with an average removal efficiency of 97%. In filter bed OSSFs, these chemicals were found to be good to moderately well removed with e.g. average removal of TCS and HHCB above 90%, whereas the remaining chemicals were between 64% and 87% [6], except TBT that achieved only 24% removal [7]. The reason for the high removal was most likely due to the hydrophobicity of the chemicals that affects their sorption potential. Chemicals with log K_{ow} higher than 4 have high sorption potentials to solids and could thus be efficiently removed [1]. For instance, the removal in soil beds was previously found to be correlated to compounds' hydrophobicity [6]. Biodegradation could also be a significant removal mechanism for certain chemicals. For instance, some biocides and biocide metabolites, including TBT, were well removed in activated soil-biofilters with biodegradation as the main removal mechanisms, showing average removal efficiencies between 82% to 100% [39], and a recent study indicated that both adsorption and biodegradation contributed to the removal of CBZ in biochar filter [40]. Field sampling protocols may influence the results as well. For example, the hydraulic retention varies between each soil bed, and it is difficult to sample effluent water that corresponds to the influent water. This may explain the low removal of TBT in the field sampling despite the good removal in the present column experiment study.

A few MPs were located close to the intersection point in the loading plot (Fig. 4B) including a rubber additive (MTBT) and some pharmaceuticals (CBZ, OZP, MTP, and LST). These compounds were better removed by Group 1 sorbents, with average removal efficiency of around 96%. Xylit and Zugol in Group 2 removed 85% of the MPs, while the inorganic sorbents could only remove 48%. The better performances of these compounds when using organic sorbents compared to inorganic materials could be explained by the influence of the functional groups present on the surface of the materials [41], the pore sizes of the sorbents (see section 3.3) and the hydrophobicity of the chemicals [6]. Surface functional groups on organic sorbents, such as GAC, usually consist of acidic and basic groups, which affect the surface charge and adsorption properties, whereas inorganic sorbents often possess surface functional groups containing metal elements [41]. The surface functional groups of GAC contribute significantly towards its adsorption ability [42]. Indeed, the better adsorption capacity of GAC sorbents over most inorganic sorbents has already been reported. For instance, the removal of several organic MPs (including multiple-class pharmaceuticals) in sand and GAC filters were comparatively assessed, and the latter exhibited higher adsorption capacity compared to sand for all tested compounds [43]. Besides, desorption of MPs from sand may occur, as was shown for pharmaceuticals temporarily retained on sand, consequently even causing negative removal efficiency [40].

A few compounds (OC, α-TPA, CF, TMDD, BAM and DF) separated from the two groups of compounds mentioned above (Fig. 4B). OC and α-TPA are quite hydrophobic chemicals and showed high removal efficiencies (median removal efficiency ≥ 90%) in a previous OSSF field sampling study [6]. Both MPs were generally well removed by most tested sorbents, as the removal efficiencies ranged from 67% to 100%. CF was poorly removed by Polonite® and Filtralite® P (15% and 0% respectively), but were well removed by other sorbents with average removal efficiencies between 62% and 97%. The removal of the surfactant (TMDD) by coal based sorbents was above 80%, Zugol® had a much lower removal efficiency which was 28%. However, inorganic sorbents showed moderate removal (47% in average). The pesticide BAM and the pharmaceutical DF were well removed by group 1 (92%) but showed almost no removal by group 2 sorbents (4%) except Xylit, which achieved 72% removal efficiency.

3.3. Impact of pore size and surface area on compound removal

The pore size of sorbents plays an important role in determining the sorption capacity of various MPs. Most of

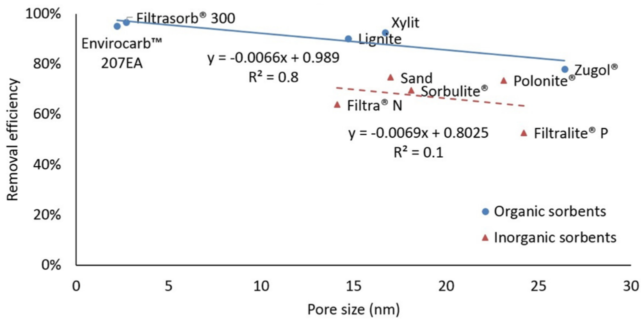

Fig. 5. Relationship between average removal efficiency of the analyzed MPs (n =19) and sorbent pore size of the tested materials (n = 10).

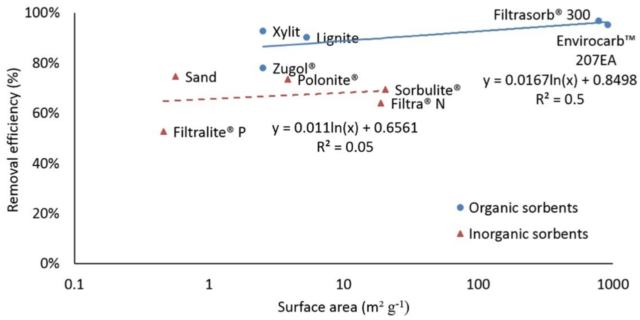

Fig. 6. Relationship between average removal efficiency of the analyzed MPs(n = 19) and sorbent surface area of the tested materials (n = 10).

the sorption takes place in the micropores (<2 nm), while mesopores (2–50 nm) and macropores (>50 nm) serve as passages for the sorbate to reach micropores [43–45]. The average pore size of Filtrasorb 300 and Envirocarb 207EA are in a beneficial range for the removal of MPs (2.7 nm and 2.2 nm; Table 1) in contrast to the other sorbents (Table 1). These two sorbents also have significantly higher fractions of pore volumes per mass unit (0.519 cm^3 g^{-1} and 0.507 cm^3 g^{-1}; Table 1), which is of essential importance for sorption. Since the functional groups of the sorbents' surface differ, also the main removal mechanisms of the MPs can differ between organic and inorganic sorbents. The impact of pore size on the removal efficiency was therefore considered separately for these two groups of sorbents. The correlation coefficient (R^2) between removal of MPs by organic sorbent and pore size was 0.8. On the contrary, the correlation coefficient between removal of MPs by inorganic sorbent and pore size was very low (0.1). The presence of small micropores is important for the removal of organic MPs from aqueous solution since the adsorption strength increased with decreasing pore size [47]. However, previous studies showed also that a coal-based activated carbon obtained a slightly better adsorption rate than a coconut-based carbon that has smaller pores attributed to a larger volume of mesopores [48]. Therefore, variation in surface properties within the sorbent appears to contribute to a good adsorption rate [44].

The total surface area may also contribute to differences between sorbents. When surface reactions dominate the sorption process, a varied surface and larger specific surface area will contribute to higher sorption rate [44,49]. For the organic sorbents, a slight tendency was observed with increasing removal efficiency by increasing surface area (Fig. 6). The two GAC materials (Filtrasorb®300 and Envirocarb™207EA), which had the largest surface areas (780 m^2 g^{-1} and 910 m^2 g^{-1}, respectively),showed the highest removal efficiencies. The other sorbents had a surface area ranging from 0.5 m^2 g^{-1} to 20 m^2 g^{-1}. Since the maximum adsorption capacity was not reached in this short-term experiment, the sorbent surface area was not a strong factor affecting the removal. For instance, Xylit (surface area 2.5 m^2 g^{-1}) achieved higher removal efficiency than lignite (surface area 5.3 m^2 g^{-1}). Moreover, Filtra® N and Sorbulite®, which had a surface area of around 20 m^2 g^{-1}, showed similar removal efficiency to Polonite® and sand (surface area 3.8 m^2 g^{-1} and 0.6 m^2 g^{-1}, respectively). Removal efficiencies and surface area were not significantly correlated (Spearman's rank correlation $R^2_{organic}$ = 0.6, $R^2_{inorganic}$ = 0.1) The lifetime of sorbents can also be influenced by the total surface area, since large surface area provides more surface functional groups involved in the interactions with MPs, which may yield a larger capacity and longer lifetime of the sorbent. Aivalioti et al. [26] showed that the adsorption capacity of BTEX, MTBE and TAME was enhanced after thermal treatment of raw lignite that increased the surface area by up to 835%.

4. Conclusions

In general, the coal-based organic sorbents performed better than the inorganic sorbents in MP removal, with on average 20% higher removal efficiency. Filtrasorb®300 and Envirocarb™207EA achieved 97% and 95% average removal, respectively. No significant differences were observed between the two types of GAC, indicating that particle size was not a relevant factor for MP removal under the conditions used in the present study. Xylit and lignite proved to have good potential to remove various MPs, with average removal efficiencies above 90%. The GACs, Xylit and lignite showed significantly higher average removal efficiency of MPs than the rest of the sorbents, while Filtralite P obtained the lowest removal efficiency of all sorbents (ANOVA, $p < 0.05$).

HCB, TCS, TBP, TDCPP, TPP, HHCB and TBT were almost totally removed by all sorbents, while BAM and DF were poorly removed by the inorganic sorbents (ANOVA, $p < 0.05$). The surface area of the organic sorbents was significantly correlated with the removal efficiency. However, the relationships between the sorbents' surface functional groups and the MPs' physicochemical properties, warrants further studies to identify molecular level understanding of the removal mechanisms.

Organic sorbents with a high calcium or iron content, e.g., Zugol®, lignite, and most inorganic sorbents (except sand) were good at removing phosphorus, while the organic sorbents Xylit, Filtrasorb®300, and Envirocarb™207EA showed low removal of phosphorus (24–66%). Ammonium-nitrogen was well removed when the pH value in the column was between 7 and 9. To achieve good removal efficiency for conventional water quality parameters as well as MPs, a combined filter system for wastewater treatments on OSSFs should be investigated.

The findings from this short-term column experiment should be followed up by a long-term column experiment and a practical field investigation.

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List of symbols

C_{in} — Influent concentration
C_{insp} — Spiked influent concentration
C_{eff} — Effluent concentration
C_{eff0} — Effluent concentration of the MP in the reference column
C_{ch} — Changed concentration of the MP in the outflow of the reference column
RE — Removal efficiency
RE_{MPs} — Removal efficiency of the MPs

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Supporting information

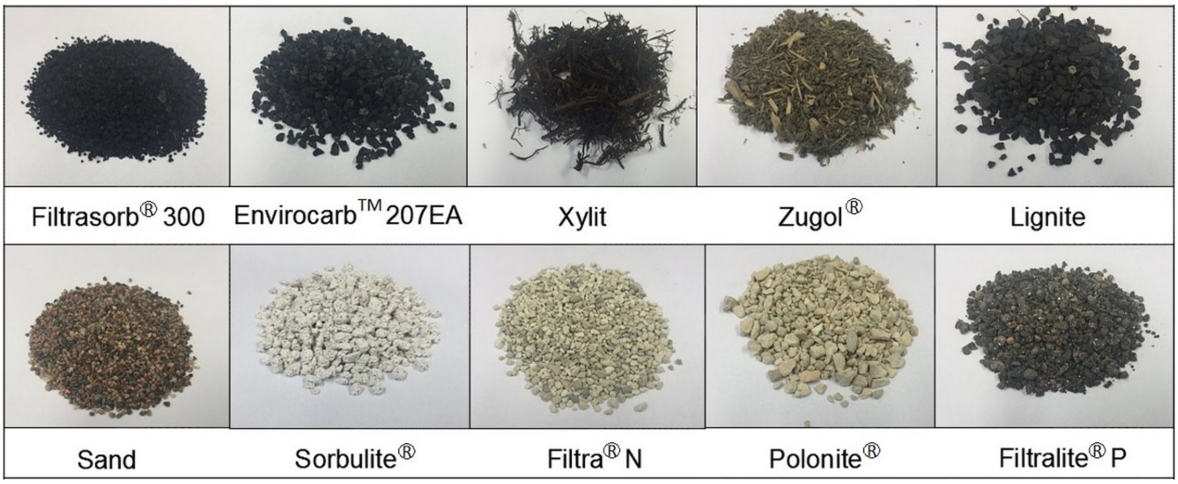

Fig. S1. Appearance of the 10 filter media selected for this study.

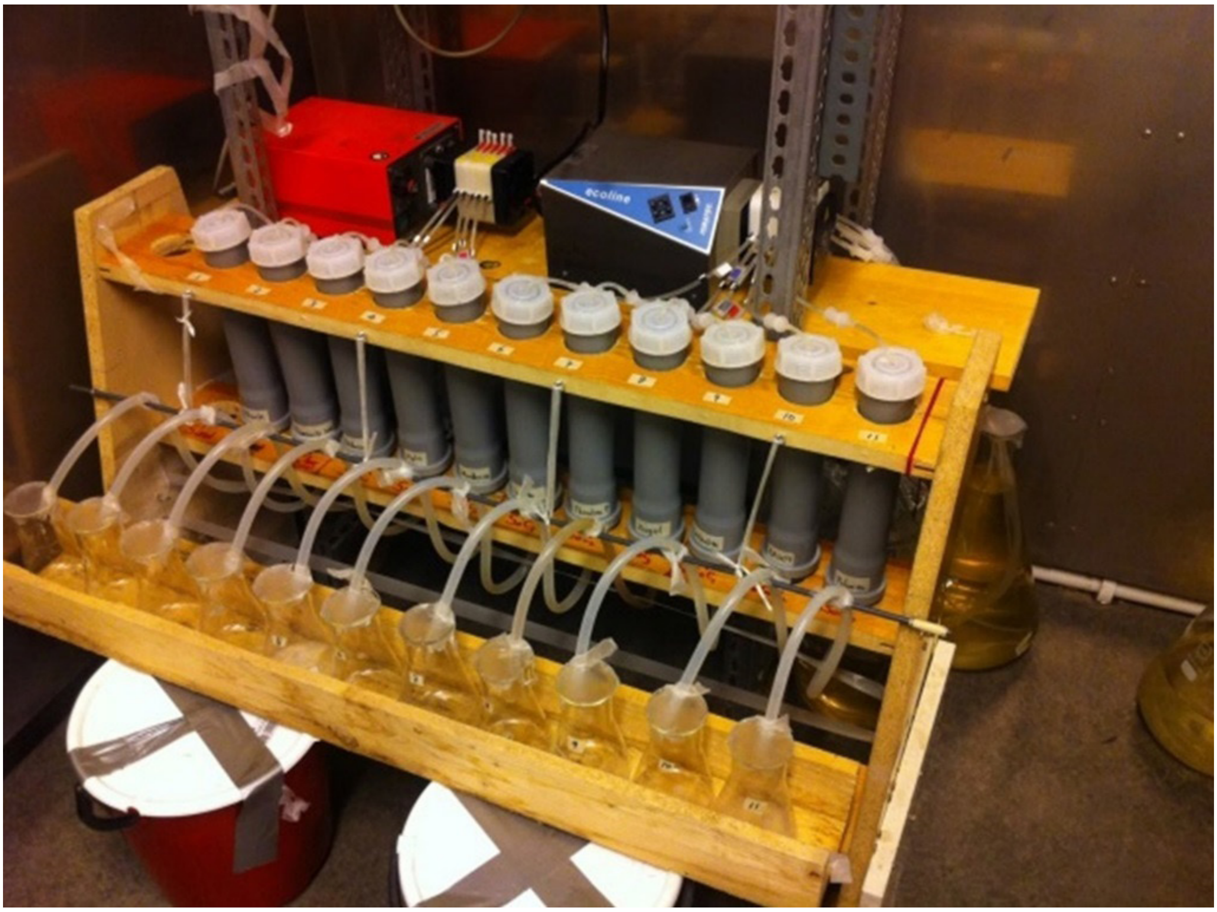

Fig. S2. Photo of the column experiment in operation.

Table S1
Native analytes and their corresponding internal standard for isotope dilution quantification

Native analyte	Corresponding internal standard
2-(Methylthio)benzothiazole	Benzothiazole-D_4
2,4,7,9-Tetramethyl-5-decyn-4,7-diol	2,4,7,9-Tetramethyl-5-decyne-4,7-diol-D_{10}
2,6-dichlorobenzamide	Isoproturon-d_6
Caffeine	
Carbamazepine	Carbamazepine-(carboxamide ^{13}C, ^{15}N)
Diclofenac	Diclofenac-$^{13}C_6$
Galaxolide	Tonalide-D_3
Hexachlorobenzene	Hexachlorobenzene-$^{13}C_6$
Losartan	Irbesartan-d_7
Metoprolol	Atenolol d7
N-Butylbenzenesulfonamide	N-Butylbenzenesulfonamide-D_9
Octocrylene	Octocrylene-D_{15}
Oxazepam	Diazepam d5
Terbutryn	Isoproturon-d_6
Tributylphosphate	Tributyl phosphate-D_{27}
Triclosan	Triclosan-D_3
Triphenylphosphate	Triphenylphosphate-D_{15}
Tris-(1,3-dichloro-2-propyl)phosphate	Tris-(1,3-dichloro-2-propyl) phosphate-D_{15}
α-Tocopheryl acetate	αTocopheryl acetate-D_9

Table S2
Concentration of MPs to the spiked 10 L feed water in μg L^{-1}

Compound	Standard mixture (μgL^{-1})	Measured concentration in the feed water (μg L^{-1})
2-(Methylthio)benzothiazole	3580	1.1
2,4,7,9-Tetramethyl-5-decyn-4,7-diol	37100	11
2,6-dichlorobenzamide	24400	12
Caffeine	21300	11
Carbamazepine	25100	13
Diclofenac	24500	12
Galaxolide	77200	23
Hexachlorobenzene	1850	0.55
Losartan	19900	9.9
Metoprolol	13900	6.9
N-Butylbenzenesulfonamide	6480	1.9
Octocrylene	116000	35
Oxazepam	19600	9.8
Terbutryn	19300	9.7
Tributylphosphate	5060	1.5
Triclosan	57000	17
Triphenylphosphate	5010	1.5
Tris-(1,3-dichloro-2-propyl)phosphate	33100	9.9
α-Tocopheryl acetate	37500	11

Table S3
Least Significant Difference (LSD) test –for individual sorbents

Tests of between-subjects effects
Dependent variable: removal

Source	Type III Sum of Squares	d_f	Mean square	F	Sig.
Corrected model	3.735[a]	9	.415	4.656	.000
Intercept	112.196	1	112.196	1258.600	.000
Filter	3.735	9	.415	4.656	.000
Error	15.154	170	.089		
Total	131.085	180			
Corrected total	18.889	179			

a. R Squared = .198 (Adjusted R Squared = .155)

Multiple comparisons
Dependent variable: removal
LSD

(I) Filter	(J) Filter	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence interval	
					Lower bound	Upper bound
Envirocarb207EA	FiltraliteP	.4372*	.09952	.000	.2408	.6337
	FiltraN	.3078*	.09952	.002	.1113	.5042
	Filtrasorb300	–.0117	.09952	.907	–.2081	.1848
	Lignite	.0433	.09952	.664	–.1531	.2398
	Polonite	.2317*	.09952	.021	.0352	.4281
	Rådasand	.2311*	.09952	.021	.0347	.4276
	Sorbulite	.2722*	.09952	.007	.0758	.4687
	Xylit	.0289	.09952	.772	–.1676	.2253
	Zugol	.2089*	.09952	.037	.0124	.4053
FiltraliteP	Envirocarb207EA	–.4372*	.09952	.000	–.6337	–.2408
	FiltraN	–.1294	.09952	.195	–.3259	.0670
	Filtrasorb300	–.4489*	.09952	.000	–.6453	–.2524
	Lignite	–.3939*	.09952	.000	–.5903	–.1974
	Polonite	–.2056*	.09952	.040	–.4020	–.0091
	Rådasand	–.2061*	.09952	.040	–.4026	–.0097
	Sorbulite	–.1650	.09952	.099	–.3615	.0315
	Xylit	–.4083*	.09952	.000	–.6048	–.2119
	Zugol	–.2283*	.09952	.023	–.4248	–.0319
FiltraN	Envirocarb207EA	–.3078*	.09952	.002	–.5042	–.1113
	FiltraliteP	.1294	.09952	.195	–.0670	.3259
	Filtrasorb300	–.3194*	.09952	.002	–.5159	–.1230
	Lignite	–.2644*	.09952	.009	–.4609	–.0680
	Polonite	–.0761	.09952	.445	–.2726	.1203
	Rådasand	–.0767	.09952	.442	–.2731	.1198
	Sorbulite	–.0356	.09952	.721	–.2320	.1609
	Xylit	–.2789*	.09952	.006	–.4753	–.0824
	Zugol	–.0989	.09952	.322	–.2953	.0976
Filtrasorb300	Envirocarb207EA	.0117	.09952	.907	–.1848	.2081
	FiltraliteP	.4489*	.09952	.000	.2524	.6453
	FiltraN	.3194*	.09952	.002	.1230	.5159
	Lignite	.0550	.09952	.581	–.1415	.2515
	Polonite	.2433*	.09952	.016	.0469	.4398
	Rådasand	.2428*	.09952	.016	.0463	.4392
	Sorbulite	.2839*	.09952	.005	.0874	.4803
	Xylit	.0406	.09952	.684	–.1559	.2370
	Zugol	.2206*	.09952	.028	.0241	.4170

(*Continued*)

Table S3 (*Continued*)

Lignite	Envirocarb207EA	–.0433	.09952	.664	–.2398	.1531
	FiltraliteP	.3939*	.09952	.000	.1974	.5903
	FiltraN	.2644*	.09952	.009	.0680	.4609
	Filtrasorb300	–.0550	.09952	.581	–.2515	.1415
	Polonite	.1883	.09952	.060	–.0081	.3848
	Rådasand	.1878	.09952	.061	–.0087	.3842
	Sorbulite	.2289*	.09952	.023	.0324	.4253
	Xylit	–.0144	.09952	.885	–.2109	.1820
	Zugol	.1656	.09952	.098	–.0309	.3620
Polonite	Envirocarb207EA	–.2317*	.09952	.021	–.4281	–.0352
	FiltraliteP	.2056*	.09952	.040	.0091	.4020
	FiltraN	.0761	.09952	.445	–.1203	.2726
	Filtrasorb300	–.2433*	.09952	.016	–.4398	–.0469
	Lignite	–.1883	.09952	.060	–.3848	.0081
	Rådasand	–.0006	.09952	.996	–.1970	.1959
	Sorbulite	.0406	.09952	.684	–.1559	.2370
	Xylit	–.2028*	.09952	.043	–.3992	–.0063
	Zugol	–.0228	.09952	.819	–.2192	.1737
Rådasand	Envirocarb207EA	–.2311*	.09952	.021	–.4276	–.0347
	FiltraliteP	.2061*	.09952	.040	.0097	.4026
	FiltraN	.0767	.09952	.442	–.1198	.2731
	Filtrasorb300	–.2428*	.09952	.016	–.4392	–.0463
	Lignite	–.1878	.09952	.061	–.3842	.0087
	Polonite	.0006	.09952	.996	–.1959	.1970
	Sorbulite	.0411	.09952	.680	–.1553	.2376
	Xylit	–.2022*	.09952	.044	–.3987	–.0058
	Zugol	–.0222	.09952	.824	–.2187	.1742
Sorbulite	Envirocarb207EA	–.2722*	.09952	.007	–.4687	–.0758
	FiltraliteP	.1650	.09952	.099	–.0315	.3615
	FiltraN	.0356	.09952	.721	–.1609	.2320
	Filtrasorb300	–.2839*	.09952	.005	–.4803	–.0874
	Lignite	–.2289*	.09952	.023	–.4253	–.0324
	Polonite	–.0406	.09952	.684	–.2370	.1559
	Rådasand	–.0411	.09952	.680	–.2376	.1553
	Xylit	–.2433*	.09952	.016	–.4398	–.0469
	Zugol	–.0633	.09952	.525	–.2598	.1331
Xylit	Envirocarb207EA	–.0289	.09952	.772	–.2253	.1676
	FiltraliteP	.4083*	.09952	.000	.2119	.6048
	FiltraN	.2789*	.09952	.006	.0824	.4753
	Filtrasorb300	–.0406	.09952	.684	–.2370	.1559
	Lignite	.0144	.09952	.885	–.1820	.2109
	Polonite	.2028*	.09952	.043	.0063	.3992
	Rådasand	.2022*	.09952	.044	.0058	.3987
	Sorbulite	.2433*	.09952	.016	.0469	.4398
	Zugol	.1800	.09952	.072	–.0165	.3765
Zugol	Envirocarb207EA	–.2089*	.09952	.037	–.4053	–.0124
	FiltraliteP	.2283*	.09952	.023	.0319	.4248
	FiltraN	.0989	.09952	.322	–.0976	.2953
	Filtrasorb300	–.2206*	.09952	.028	–.4170	–.0241
	Lignite	–.1656	.09952	.098	–.3620	.0309
	Polonite	.0228	.09952	.819	–.1737	.2192
	Rådasand	.0222	.09952	.824	–.1742	.2187
	Sorbulite	.0633	.09952	.525	–.1331	.2598
	Xylit	–.1800	.09952	.072	–.3765	.0165

Based on observed means.
The error term is Mean Square (Error) = .089.
*. The mean difference is significant at the 0.05 level.

Table S4
Least significant difference (LSD) test – for individual MPs

Tests of between-subjects effects					
Dependent variable: RE					
Source	Type III Sum of Squares	d_f	Mean square	F	Sig.
Corrected model	6.957[a]	17	.409	5.558	.000
Intercept	112.112	1	112.112	1522.573	.000
MPs	6.957	17	.409	5.558	.000
Error	11.929	162	.074		
Total	130.997	180			
Corrected total	18.886	179			

a. R Squared = .368 (Adjusted R Squared = .302)

Multiple comparisons						
Dependent variable: RE						
LSD						
(I) MPs	(J) MPs	Mean difference (I-J)	Std. error	Sig.	95% Confidence interval	
					Lower bound	Upper bound
aTPA	BAM	.487720*	.1213533	.000	.248082	.727358
	CBZ	.204360	.1213533	.094	–.035278	.443998
	CF	.123450	.1213533	.311	–.116188	.363088
	DF	.452280*	.1213533	.000	.212642	.691918
	HCB	–.158640	.1213533	.193	–.398278	.080998
	HHCB	–.158290	.1213533	.194	–.397928	.081348
	LST	.187110	.1213533	.125	–.052528	.426748
	MEP	.021060	.1213533	.862	–.218578	.260698
	MTBT	.109350	.1213533	.369	–.130288	.348988
	OC	–.063190	.1213533	.603	–.302828	.176448
	OZP	.126430	.1213533	.299	–.113208	.366068
	TBP	–.095890	.1213533	.431	–.335528	.143748
	TBT	–.120040	.1213533	.324	–.359678	.119598
	TCS	–.114860	.1213533	.345	–.354498	.124778
	TDCPP	–.139090	.1213533	.253	–.378728	.100548
	TMDD	.223630	.1213533	.067	–.016008	.463268
	TPP	–.146580	.1213533	.229	–.386218	.093058
BAM	aTPA	–.487720*	.1213533	.000	–.727358	–.248082
	CBZ	–.283360*	.1213533	.021	–.522998	–.043722
	CF	–.364270*	.1213533	.003	–.603908	–.124632
	DF	–.035440	.1213533	.771	–.275078	.204198
	HCB	–.646360*	.1213533	.000	–.885998	–.406722
	HHCB	–.646010*	.1213533	.000	–.885648	–.406372
	LST	–.300610*	.1213533	.014	–.540248	–.060972
	MEP	–.466660*	.1213533	.000	–.706298	–.227022
	MTBT	–.378370*	.1213533	.002	–.618008	–.138732
	OC	–.550910*	.1213533	.000	–.790548	–.311272
	OZP	–.361290*	.1213533	.003	–.600928	–.121652
	TBP	–.583610*	.1213533	.000	–.823248	–.343972
	TBT	–.607760*	.1213533	.000	–.847398	–.368122
	TCS	–.602580*	.1213533	.000	–.842218	–.362942
	TDCPP	–.626810*	.1213533	.000	–.866448	–.387172
	TMDD	–.264090*	.1213533	.031	–.503728	–.024452
	TPP	–.634300*	.1213533	.000	–.873938	–.394662

(*Continued*)

Table S4 (*Continued*)

CBZ	aTPA	–.204360	.1213533	.094	–.443998	.035278
	BAM	.283360*	.1213533	.021	.043722	.522998
	CF	–.080910	.1213533	.506	–.320548	.158728
	DF	.247920*	.1213533	.043	.008282	.487558
	HCB	–.363000*	.1213533	.003	–.602638	–.123362
	HHCB	–.362650*	.1213533	.003	–.602288	–.123012
	LST	–.017250	.1213533	.887	–.256888	.222388
	MEP	–.183300	.1213533	.133	–.422938	.056338
	MTBT	–.095010	.1213533	.435	–.334648	.144628
	OC	–.267550*	.1213533	.029	–.507188	–.027912
	OZP	–.077930	.1213533	.522	–.317568	.161708
	TBP	–.300250*	.1213533	.014	–.539888	–.060612
	TBT	–.324400*	.1213533	.008	–.564038	–.084762
	TCS	–.319220*	.1213533	.009	–.558858	–.079582
	TDCPP	–.343450*	.1213533	.005	–.583088	–.103812
	TMDD	.019270	.1213533	.874	–.220368	.258908
	TPP	–.350940*	.1213533	.004	–.590578	–.111302
CF	aTPA	–.123450	.1213533	.311	–.363088	.116188
	BAM	.364270*	.1213533	.003	.124632	.603908
	CBZ	.080910	.1213533	.506	–.158728	.320548
	DF	.328830*	.1213533	.007	.089192	.568468
	HCB	–.282090*	.1213533	.021	–.521728	–.042452
	HHCB	–.281740*	.1213533	.021	–.521378	–.042102
	LST	.063660	.1213533	.601	–.175978	.303298
	MEP	–.102390	.1213533	.400	–.342028	.137248
	MTBT	–.014100	.1213533	.908	–.253738	.225538
	OC	–.186640	.1213533	.126	–.426278	.052998
	OZP	.002980	.1213533	.980	–.236658	.242618
	TBP	–.219340	.1213533	.073	–.458978	.020298
	TBT	–.243490*	.1213533	.046	–.483128	–.003852
	TCS	–.238310	.1213533	.051	–.477948	.001328
	TDCPP	–.262540*	.1213533	.032	–.502178	–.022902
	TMDD	.100180	.1213533	.410	–.139458	.339818
	TPP	–.270030*	.1213533	.027	–.509668	–.030392
DF	aTPA	–.452280*	.1213533	.000	–.691918	–.212642
	BAM	.035440	.1213533	.771	–.204198	.275078
	CBZ	–.247920*	.1213533	.043	–.487558	–.008282
	CF	–.328830*	.1213533	.007	–.568468	–.089192
	HCB	–.610920*	.1213533	.000	–.850558	–.371282
	HHCB	–.610570*	.1213533	.000	–.850208	–.370932
	LST	–.265170*	.1213533	.030	–.504808	–.025532
	MEP	–.431220*	.1213533	.000	–.670858	–.191582
	MTBT	–.342930*	.1213533	.005	–.582568	–.103292
	OC	–.515470*	.1213533	.000	–.755108	–.275832
	OZP	–.325850*	.1213533	.008	–.565488	–.086212
	TBP	–.548170*	.1213533	.000	–.787808	–.308532
	TBT	–.572320*	.1213533	.000	–.811958	–.332682
	TCS	–.567140*	.1213533	.000	–.806778	–.327502
	TDCPP	–.591370*	.1213533	.000	–.831008	–.351732
	TMDD	–.228650	.1213533	.061	–.468288	.010988
	TPP	–.598860*	.1213533	.000	–.838498	–.359222

(*Continued*)

Table S4 (*Continued*)

HCB	aTPA	.158640	.1213533	.193	–.080998	.398278
	BAM	.646360*	.1213533	.000	.406722	.885998
	CBZ	.363000*	.1213533	.003	.123362	.602638
	CF	.282090*	.1213533	.021	.042452	.521728
	DF	.610920*	.1213533	.000	.371282	.850558
	HHCB	.000350	.1213533	.998	–.239288	.239988
	LST	.345750*	.1213533	.005	.106112	.585388
	MEP	.179700	.1213533	.141	–.059938	.419338
	MTBT	.267990*	.1213533	.029	.028352	.507628
	OC	.095450	.1213533	.433	–.144188	.335088
	OZP	.285070*	.1213533	.020	.045432	.524708
	TBP	.062750	.1213533	.606	–.176888	.302388
	TBT	.038600	.1213533	.751	–.201038	.278238
	TCS	.043780	.1213533	.719	–.195858	.283418
	TDCPP	.019550	.1213533	.872	–.220088	.259188
	TMDD	.382270*	.1213533	.002	.142632	.621908
	TPP	.012060	.1213533	.921	–.227578	.251698
HHCB	aTPA	.158290	.1213533	.194	–.081348	.397928
	BAM	.646010*	.1213533	.000	.406372	.885648
	CBZ	.362650*	.1213533	.003	.123012	.602288
	CF	.281740*	.1213533	.021	.042102	.521378
	DF	.610570*	.1213533	.000	.370932	.850208
	HCB	–.000350	.1213533	.998	–.239988	.239288
	LST	.345400*	.1213533	.005	.105762	.585038
	MEP	.179350	.1213533	.141	–.060288	.418988
	MTBT	.267640*	.1213533	.029	.028002	.507278
	OC	.095100	.1213533	.434	–.144538	.334738
	OZP	.284720*	.1213533	.020	.045082	.524358
	TBP	.062400	.1213533	.608	–.177238	.302038
	TBT	.038250	.1213533	.753	–.201388	.277888
	TCS	.043430	.1213533	.721	–.196208	.283068
	TDCPP	.019200	.1213533	.874	–.220438	.258838
	TMDD	.381920*	.1213533	.002	.142282	.621558
	TPP	.011710	.1213533	.923	–.227928	.251348
LST	aTPA	–.187110	.1213533	.125	–.426748	.052528
	BAM	.300610*	.1213533	.014	.060972	.540248
	CBZ	.017250	.1213533	.887	–.222388	.256888
	CF	–.063660	.1213533	.601	–.303298	.175978
	DF	.265170*	.1213533	.030	.025532	.504808
	HCB	–.345750*	.1213533	.005	–.585388	–.106112
	HHCB	–.345400*	.1213533	.005	–.585038	–.105762
	MEP	–.166050	.1213533	.173	–.405688	.073588
	MTBT	–.077760	.1213533	.523	–.317398	.161878
	OC	–.250300*	.1213533	.041	–.489938	–.010662
	OZP	–.060680	.1213533	.618	–.300318	.178958
	TBP	–.283000*	.1213533	.021	–.522638	–.043362
	TBT	–.307150*	.1213533	.012	–.546788	–.067512
	TCS	–.301970*	.1213533	.014	–.541608	–.062332
	TDCPP	–.326200*	.1213533	.008	–.565838	–.086562
	TMDD	.036520	.1213533	.764	–.203118	.276158
	TPP	–.333690*	.1213533	.007	–.573328	–.094052

(*Continued*)

Table S4 (*Continued*)

MEP	aTPA	–.021060	.1213533	.862	–.260698	.218578
	BAM	.466660*	.1213533	.000	.227022	.706298
	CBZ	.183300	.1213533	.133	–.056338	.422938
	CF	.102390	.1213533	.400	–.137248	.342028
	DF	.431220*	.1213533	.000	.191582	.670858
	HCB	–.179700	.1213533	.141	–.419338	.059938
	HHCB	–.179350	.1213533	.141	–.418988	.060288
	LST	.166050	.1213533	.173	–.073588	.405688
	MTBT	.088290	.1213533	.468	–.151348	.327928
	OC	–.084250	.1213533	.489	–.323888	.155388
	OZP	.105370	.1213533	.387	–.134268	.345008
	TBP	–.116950	.1213533	.337	–.356588	.122688
	TBT	–.141100	.1213533	.247	–.380738	.098538
	TCS	–.135920	.1213533	.264	–.375558	.103718
	TDCPP	–.160150	.1213533	.189	–.399788	.079488
	TMDD	.202570	.1213533	.097	–.037068	.442208
	TPP	–.167640	.1213533	.169	–.407278	.071998
MTBT	aTPA	–.109350	.1213533	.369	–.348988	.130288
	BAM	.378370*	.1213533	.002	.138732	.618008
	CBZ	.095010	.1213533	.435	–.144628	.334648
	CF	.014100	.1213533	.908	–.225538	.253738
	DF	.342930*	.1213533	.005	.103292	.582568
	HCB	–.267990*	.1213533	.029	–.507628	–.028352
	HHCB	–.267640*	.1213533	.029	–.507278	–.028002
	LST	.077760	.1213533	.523	–.161878	.317398
	MEP	–.088290	.1213533	.468	–.327928	.151348
	OC	–.172540	.1213533	.157	–.412178	.067098
	OZP	.017080	.1213533	.888	–.222558	.256718
	TBP	–.205240	.1213533	.093	–.444878	.034398
	TBT	–.229390	.1213533	.061	–.469028	.010248
	TCS	–.224210	.1213533	.066	–.463848	.015428
	TDCPP	–.248440*	.1213533	.042	–.488078	–.008802
	TMDD	.114280	.1213533	.348	–.125358	.353918
	TPP	–.255930*	.1213533	.036	–.495568	–.016292
OC	aTPA	.063190	.1213533	.603	–.176448	.302828
	BAM	.550910*	.1213533	.000	.311272	.790548
	CBZ	.267550*	.1213533	.029	.027912	.507188
	CF	.186640	.1213533	.126	–.052998	.426278
	DF	.515470*	.1213533	.000	.275832	.755108
	HCB	–.095450	.1213533	.433	–.335088	.144188
	HHCB	–.095100	.1213533	.434	–.334738	.144538
	LST	.250300*	.1213533	.041	.010662	.489938
	MEP	.084250	.1213533	.489	–.155388	.323888
	MTBT	.172540	.1213533	.157	–.067098	.412178
	OZP	.189620	.1213533	.120	–.050018	.429258
	TBP	–.032700	.1213533	.788	–.272338	.206938
	TBT	–.056850	.1213533	.640	–.296488	.182788
	TCS	–.051670	.1213533	.671	–.291308	.187968
	TDCPP	–.075900	.1213533	.533	–.315538	.163738
	TMDD	.286820*	.1213533	.019	.047182	.526458
	TPP	–.083390	.1213533	.493	–.323028	.156248

(*Continued*)

Table S4 (*Continued*)

OZP	aTPA	–.126430	.1213533	.299	–.366068	.113208
	BAM	.361290*	.1213533	.003	.121652	.600928
	CBZ	.077930	.1213533	.522	–.161708	.317568
	CF	–.002980	.1213533	.980	–.242618	.236658
	DF	.325850*	.1213533	.008	.086212	.565488
	HCB	–.285070*	.1213533	.020	–.524708	–.045432
	HHCB	–.284720*	.1213533	.020	–.524358	–.045082
	LST	.060680	.1213533	.618	–.178958	.300318
	MEP	–.105370	.1213533	.387	–.345008	.134268
	MTBT	–.017080	.1213533	.888	–.256718	.222558
	OC	–.189620	.1213533	.120	–.429258	.050018
	TBP	–.222320	.1213533	.069	–.461958	.017318
	TBT	–.246470*	.1213533	.044	–.486108	–.006832
	TCS	–.241290*	.1213533	.048	–.480928	–.001652
	TDCPP	–.265520*	.1213533	.030	–.505158	–.025882
	TMDD	.097200	.1213533	.424	–.142438	.336838
	TPP	–.273010*	.1213533	.026	–.512648	–.033372
TBP	aTPA	.095890	.1213533	.431	–.143748	.335528
	BAM	.583610*	.1213533	.000	.343972	.823248
	CBZ	.300250*	.1213533	.014	.060612	.539888
	CF	.219340	.1213533	.073	–.020298	.458978
	DF	.548170*	.1213533	.000	.308532	.787808
	HCB	–.062750	.1213533	.606	–.302388	.176888
	HHCB	–.062400	.1213533	.608	–.302038	.177238
	LST	.283000*	.1213533	.021	.043362	.522638
	MEP	.116950	.1213533	.337	–.122688	.356588
	MTBT	.205240	.1213533	.093	–.034398	.444878
	OC	.032700	.1213533	.788	–.206938	.272338
	OZP	.222320	.1213533	.069	–.017318	.461958
	TBT	–.024150	.1213533	.843	–.263788	.215488
	TCS	–.018970	.1213533	.876	–.258608	.220668
	TDCPP	–.043200	.1213533	.722	–.282838	.196438
	TMDD	.319520*	.1213533	.009	.079882	.559158
	TPP	–.050690	.1213533	.677	–.290328	.188948
TBT	aTPA	.120040	.1213533	.324	–.119598	.359678
	BAM	.607760*	.1213533	.000	.368122	.847398
	CBZ	.324400*	.1213533	.008	.084762	.564038
	CF	.243490*	.1213533	.046	.003852	.483128
	DF	.572320*	.1213533	.000	.332682	.811958
	HCB	–.038600	.1213533	.751	–.278238	.201038
	HHCB	–.038250	.1213533	.753	–.277888	.201388
	LST	.307150*	.1213533	.012	.067512	.546788
	MEP	.141100	.1213533	.247	–.098538	.380738
	MTBT	.229390	.1213533	.061	–.010248	.469028
	OC	.056850	.1213533	.640	–.182788	.296488
	OZP	.246470*	.1213533	.044	.006832	.486108
	TBP	.024150	.1213533	.843	–.215488	.263788
	TCS	.005180	.1213533	.966	–.234458	.244818
	TDCPP	–.019050	.1213533	.875	–.258688	.220588
	TMDD	.343670*	.1213533	.005	.104032	.583308
	TPP	–.026540	.1213533	.827	–.266178	.213098

(*Continued*)

Table S4 (*Continued*)

TCS	aTPA	.114860	.1213533	.345	–.124778	.354498
	BAM	.602580*	.1213533	.000	.362942	.842218
	CBZ	.319220*	.1213533	.009	.079582	.558858
	CF	.238310	.1213533	.051	–.001328	.477948
	DF	.567140*	.1213533	.000	.327502	.806778
	HCB	–.043780	.1213533	.719	–.283418	.195858
	HHCB	–.043430	.1213533	.721	–.283068	.196208
	LST	.301970*	.1213533	.014	.062332	.541608
	MEP	.135920	.1213533	.264	–.103718	.375558
	MTBT	.224210	.1213533	.066	–.015428	.463848
	OC	.051670	.1213533	.671	–.187968	.291308
	OZP	.241290*	.1213533	.048	.001652	.480928
	TBP	.018970	.1213533	.876	–.220668	.258608
	TBT	–.005180	.1213533	.966	–.244818	.234458
	TDCPP	–.024230	.1213533	.842	–.263868	.215408
	TMDD	.338490*	.1213533	.006	.098852	.578128
	TPP	–.031720	.1213533	.794	–.271358	.207918
TDCPP	aTPA	.139090	.1213533	.253	–.100548	.378728
	BAM	.626810*	.1213533	.000	.387172	.866448
	CBZ	.343450*	.1213533	.005	.103812	.583088
	CF	.262540*	.1213533	.032	.022902	.502178
	DF	.591370*	.1213533	.000	.351732	.831008
	HCB	–.019550	.1213533	.872	–.259188	.220088
	HHCB	–.019200	.1213533	.874	–.258838	.220438
	LST	.326200*	.1213533	.008	.086562	.565838
	MEP	.160150	.1213533	.189	–.079488	.399788
	MTBT	.248440*	.1213533	.042	.008802	.488078
	OC	.075900	.1213533	.533	–.163738	.315538
	OZP	.265520*	.1213533	.030	.025882	.505158
	TBP	.043200	.1213533	.722	–.196438	.282838
	TBT	.019050	.1213533	.875	–.220588	.258688
	TCS	.024230	.1213533	.842	–.215408	.263868
	TMDD	.362720*	.1213533	.003	.123082	.602358
	TPP	–.007490	.1213533	.951	–.247128	.232148
TMDD	aTPA	–.223630	.1213533	.067	–.463268	.016008
	BAM	.264090*	.1213533	.031	.024452	.503728
	CBZ	–.019270	.1213533	.874	–.258908	.220368
	CF	–.100180	.1213533	.410	–.339818	.139458
	DF	.228650	.1213533	.061	–.010988	.468288
	HCB	–.382270*	.1213533	.002	–.621908	–.142632
	HHCB	–.381920*	.1213533	.002	–.621558	–.142282
	LST	–.036520	.1213533	.764	–.276158	.203118
	MEP	–.202570	.1213533	.097	–.442208	.037068
	MTBT	–.114280	.1213533	.348	–.353918	.125358
	OC	–.286820*	.1213533	.019	–.526458	–.047182
	OZP	–.097200	.1213533	.424	–.336838	.142438
	TBP	–.319520*	.1213533	.009	–.559158	–.079882
	TBT	–.343670*	.1213533	.005	–.583308	–.104032
	TCS	–.338490*	.1213533	.006	–.578128	–.098852
	TDCPP	–.362720*	.1213533	.003	–.602358	–.123082
	TPP	–.370210*	.1213533	.003	–.609848	–.130572

(*Continued*)

Table S4 (*Continued*)

TPP	aTPA	.146580	.1213533	.229	–.093058	.386218
	BAM	.634300*	.1213533	.000	.394662	.873938
	CBZ	.350940*	.1213533	.004	.111302	.590578
	CF	.270030*	.1213533	.027	.030392	.509668
	DF	.598860*	.1213533	.000	.359222	.838498
	HCB	–.012060	.1213533	.921	–.251698	.227578
	HHCB	–.011710	.1213533	.923	–.251348	.227928
	LST	.333690*	.1213533	.007	.094052	.573328
	MEP	.167640	.1213533	.169	–.071998	.407278
	MTBT	.255930*	.1213533	.036	.016292	.495568
	OC	.083390	.1213533	.493	–.156248	.323028
	OZP	.273010*	.1213533	.026	.033372	.512648
	TBP	.050690	.1213533	.677	–.188948	.290328
	TBT	.026540	.1213533	.827	–.213098	.266178
	TCS	.031720	.1213533	.794	–.207918	.271358
	TDCPP	.007490	.1213533	.951	–.232148	.247128
	TMDD	.370210*	.1213533	.003	.130572	.609848

Based on observed means.

The error term is Mean Square(Error) = .074.

*. The mean difference is significant at the 0.05 level.