Adsorption of Organic Pollutants in Stormwater: Evaluation of Four Potential Sorbents

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ABSTRACT

Adsorption filter is one of the most promising techniques used for removal of dissolved, colloidal and particulate pollutants from stormwater. The aim of this study was to compare the capacity of four filter materials—zeolite, cellulose, polypropylene/polyethylene (PP/PE) fibres and pine bark to adsorb organic pollutants frequently detected in stormwater. In batch tests, synthetic stormwater (spiked with polycyclic aromatic hydrocarbons [PAHs], alkyl phenols and phthalates) was kept with media for 24 hours. The compounds were then liquid–liquid extracted and analysed using gas chromatography–mass spectrometry (GC-MS). Zeolite and cellulose showed very low sorption capacity for the organic contaminants. More than 70% of the initial concentration of all tested compounds was removed using PP/PE fibres and >80% with pine bark. The highest adsorption capacity was found for PAHs (up to 44 µg/g) using PP/PE fibres and bark. For all tested compounds, maximum adsorption was obtained within 30 minutes of using these materials. Future research using natural stormwater should investigate the effect of colloidal transport of pollutants through PP/PE fibres and pine bark and the materials' capacities to adsorb other pollutants, including metals.

KEYWORDS
Adsorption filter, stormwater treatment, priority pollutants, mineral and wood-based media

INTRODUCTION
Stormwaterhasbeen found to containorganic pollutants, including polycyclic aromatic hydrocarbons (PAHs), alkylphenols and phthalates,at levelsthat oftenexceed national and international environmental quality standards (Björklund *et al.* 2009; 2011; Zgheib *et al.* 2011).Treatment ofstormwateris therefore an important stepto achievethe objectives for water quality. Although many organic pollutants should, in theory, bindto particlesandberemoved, e.g. through sedimentation, previousstudies have shown thatthe pollutants are often found in the dissolvedphaseincontaminatedwater(Kalmykova *et al.*2013; Zgheib *et al.* 2011).Filterswithadsorbing mediahave the potential to removebothdissolvedandparticle-bound organic contaminants and metals from stormwater.

The aim of this studywas tocompare the adsorption capacity ofa variety offilter mediafor organic pollutants frequently detected in stormwater. The goal was to identify materials that have high potential use in filters for stormwater treatment. Materials thatare inexpensiveand abundant,may bereused or recycled, and haveadequatepermeability to waterwere selected.

The study focussed on seven organic pollutants – three PAHs (fluorene [FL], anthracene [ANT] and pyrene [PYR]), two alkylphenols (nonyl- [NP] and octylphenol [OP]) and two phthalates (dibutyl [DBP] and di(2-ethylhexyl) phthalate [DEHP])–frequently detected in stormwater, but not previouslystudiedin this regard. Batch tests were performed to study the adsorption kinetics and the adsorption capacities of the materials at equilibrium.

MATERIALS AND METHODS
Sorbents
Fourmaterials were selected for analysis; the mineral zeolite(Bear River Zeolite, USA), cellulose pellets(by-product from pulp and paper industry), fibres made from polypropylene (PP) and polyethylene (PE) (waste product from plastics industry),and pine bark (Ecobark, Nyman Consulting, Sweden). All media are commercially available and have previously been used for sorption of metals, oils, acids or solvents. The mineral and bark were sieved to the desired particle size (0.6–2 mm), washed and dried at 105°C before use. The organic content of the materials was determined by loss on ignition (LOI). The specific surface area was determined by the BET nitrogen adsorption method, using a FlowSorb II 2300 surface analyzer (Micromeritics).

Leaching of Dissolved Organic Carbon and Organic Pollutants from Raw Materials
One gram of each material (0.1 g of the cellulose, due to its expansion in water) was mixed with 50 mL Milli-Q water for 24 h. The samples were filtered through a 0.45 μm cellulose nitrate filter (Millipore) and analyzed for leached dissolved organic carbon (DOC) using a Lachat Instrument IL 500 TOC analyzer. For analysis of leached organic compounds from media, 3 g of each material (0.3 g cellulose) was mixed with 150 mL Milli-Q water for 24 h.

Adsorption Tests
Adsorption was studied through batch testswith syntheticstormwatercontainingdissolved organic material (DOM), here humic acids (HAs), spiked witha mixture of organic compounds. The sample composition is a simplification of natural conditionswherepollutantscoexistandwhereDOMcan affectthe solubility,and thereforethe adsorption, of organic pollutants and metals (Brown and Peake 2003; Kalmykova *et al.* 2010). A stock solution was prepared by dissolving Fluka humic acid in Milli-Q water. The samples were spiked with the HA stock to 20 mg DOC/L, typical of what is found in natural stormwater samples, and pH was adjusted to 7. 150 mL HA solution was then spiked at five different concentrations (10–300 μg/L) with organic compounds. 1.0 g of each material (0.10 g of cellulose) was then added to the spiked solution and shaken, at room temperature, for 24 h. Samples were then centrifuged at 2000 rpm for 10 min, and those with bark were also sieved through a stainless steel mesh (No. 100). The samples for adsorption kinetics tests were spiked to 100 μg/L with organic compounds and mixed for 10, 20, 30, 60 and 120 min. For each setof tests, a matrix blank and a matrix spike were prepared, following the above procedure, to determine contamination and loss of analytes, respectively. All samples were prepared in duplicate.

Sample Extraction and Analysis
All compounds were simultaneously liquid-liquid extracted with 3×25 mL dichloromethane. The extracts were evaporated using a rotary evaporator and then further concentrated under a stream of N_2. The samples were reconstituted with 1.0 mL toluene and internal standards(9-chloroanthracene and phenanthrene-d_{10})were added before analysis using a 6890 HP/Agilent gas chromatograph (30 m×0.25 mm I.D. DB-5 column from J&W Scientific, Folsom, USA)

with a 6890 series injector and a quadrupole 5973 mass selective detector from Agilent Technologies, Wilmington, USA.

Data Analysis
The program IBM SPSS Statistics 20 was employed to perform all statistical analyses.

The adsorption capacity at equilibrium, q_e (µg/g), was calculated using:
$$q_e = V \times \frac{C_0 - C_e}{m}$$
Where C_0 and C_e are the initial and equilibrium concentrations (µg/L) of the organic compounds in solution, respectively; V is the volume of the solution (L) and m is the weight of the adsorbent (g).

Linearized forms of both the Freundlich and the Langmuir models were used to study the adsorption isotherms of PP/PE fibres and pine bark.
$$\log q_e = \log K_F + \frac{1}{n} \log C_e \qquad \text{(Freundlich)}$$
$$\frac{1}{q_e} = \frac{1}{q_{max}} + \frac{1}{K_L \times q_{max}} \times \frac{1}{C_e} \qquad \text{(Langmuir)}$$
Where q_e is the amount of adsorbed analytes onto adsorbent at equilibrium (µg/g); C_e is the equilibrium concentration of analytes remaining in solution (µg/L); K_F ((µg/g)(L/µg)$^{1/n}$), n and K_L (L/µg) are adsorption system specific constants; and q_{max} is the maximum adsorption capacity (µg/g). The isotherm constants can be evaluated from linear plots of the equations.

Commonly used kinetic models to evaluate the adsorption rate are the Lagergren pseudo-first-order adsorption rate equation and the pseudo-second order equation:
$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \qquad \text{(pseudo-firstorder)}$$
$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \qquad \text{(pseudo-second order)}$$
Where q_e and q_t are the amounts of adsorbed analytes (µg/g) onto adsorbent at equilibrium and at time t (min), respectively; k_1 (1/min) is the rate constant for first-order sorption; k_2 (g/(µg min)) is the rate constant of second-order sorption. The best fitting model was examined by plotting $\log(q_e - q_t)$ vs t and (t/q_t) vs t and comparing corresponding linear regression correlation coefficients for each compound. Based on the t/q vs t plot, the intercept and the slope were used to calculate the rate constant k_2 and the equilibrium adsorption q_e for the pseudo-secondordermodel.
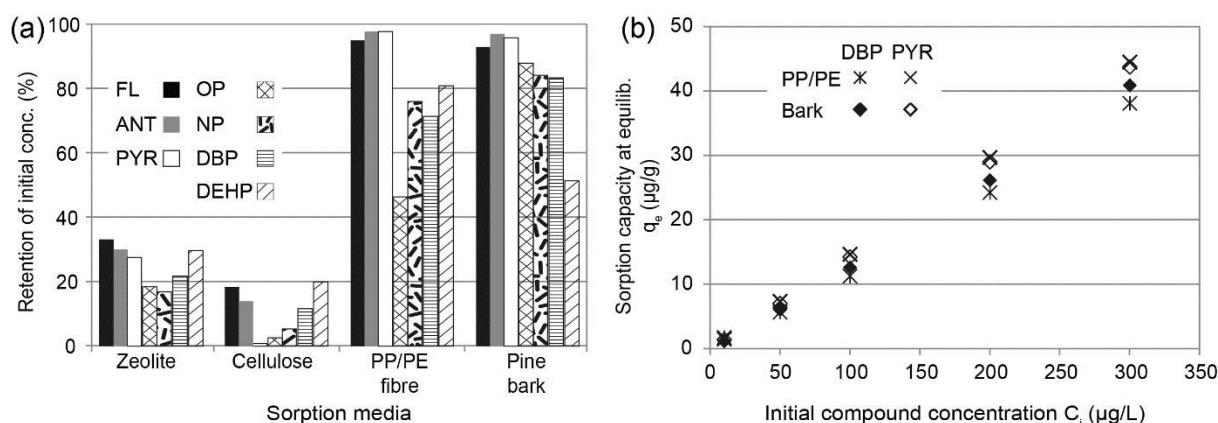
RESULTS AND DISCUSSION
Characteristics of the Materials
The zeolite tested is a crushed clinoptilolite(85%). The cellulose shows high hydrophilicity and expands considerably in water. The PP/PE is made up of up to 200-mm-long, very thin fibres susceptible to wetting. The bark is hydrophobic and resists wetting up to 2 h of soaking. The organic media show considerably lower BET surface area (<1.4 m^2/g) and higher organic content (cellulose87%; PP/PE 91%; bark 99%) than the mineral (34 m^2/g and 1.7%, respectively).

Adsorption of Humic Acids and Leaching from Raw Materials
Neither of the tested materials adsorbed humic acids to a substantialdegree (>5 mg/L), nor were any of the tested organic compounds desorbed in detectable amounts from the raw materials. However, background contamination (<4 µg/L) of the two phthalates could be

detected in most samples. Zeolite, cellulose and the bark exhibit negative surface charge (Pitcher *et al.* 2004; Wan Ngah and Hanafiah 2008), as do humic acids at neutral pH. Hence limited adsorption of HA occurs. The pine bark desorbs DOC up to ~100 mg/L, exceeding levels found in natural stormwater (typically 20–30 mg DOC/L). Kalmykova *et al.* (2010) showed that DOC leaching from a peat column fed with a metal solution correlated significantly with metal concentrations in the column effluent in the initial stage of their experiment. The metal transport was partly attributed to metals forming colloidal organic complexes with easily soluble organic matter from the peat. In addition, Kalmykova *et al.* (2014) showed that humic colloids and attached organic contaminants are not efficiently adsorbed by peat filters. As humics contain both non-polar and polar surface groups, many organic compounds are expected to bind to humic colloids (Kalmykova *et al.* 2014). These studies indicate that if metals and organic pollutants are attached to humic colloids, the colloids may negatively affect the pollutants' adsorption onto e.g. wood-based media, which, similarly to peat, exhibit the same surface charge as the humics.

Figure 1. a) Retention (average of five tested concentrations) of fluorene (FL), anthracene (ANT), pyrene (PYR), octylphenol (OP), nonylphenol (OP), dibutyl (DBP) and di(2-ethylhexyl) phthalate (DEHP) after 24 h; b) Adsorption capacity of DBP and PYR after 24 h using PP/PE fibres and pine bark. Similar trends in adsorption capacities were obtained for other tested compounds (not shown).

Removal of Organic Compounds

The removal capacities of zeolite and cellulose for the organic contaminants were very low (Figure 1a). In fact, there was no significant difference in adsorption of compounds using zeolite (M=67, SD=18) and compound losses during extraction and analysis (M=70, SD=8), t(14)=0.28, p=.783,and betweenadsorption on cellulose (M=76, SD=16) and compound losses (MD=82, SD=18), t(14)=0.69, p=.500 (independent samples t-test). These results were expected, as minerals have a negatively charged surface and may remove ions frompolluted waters by ionic exchange processes; thus they are better suited for metal adsorption (Pitcher *et al.* 2004; Seelsaen *et al.* 2006). The low adsorption capacity of cellulose for organic contaminants is due to its hydrophilicity and negative surface charge, which promote sorption of cations (Wan Ngah and Hanafiah 2008).

In general, over 70% of the initial concentration of all tested compounds was removed using PP/PE fibres, and over 80% using pine bark(Figure 1a). For all compounds, the removed amount was either positively correlated with initial concentrations or constant over the spiked concentration range. The PAHs were removed to the highest degree (92–97%) by both PP/PE

fibres and bark(Figure 1a). The average removal of NP, OP and DBP exceeded 80% using pine bark, while the average removal byPP/PE was around 10% lower. However, only 50% of the initial OP concentration was removed using PP/PE fibres, and the average removal of DEHP was ~50 and 80% for pine bark and PP/PE fibres, respectively. The generally high removals indicate that, despite possible colloid-formation, humic acids did not have asubstantial negative effect on the adsorption of organic pollutants in this study. This is true even for the wood-based material, which is not expected to adsorb humic colloids and thereto attached contaminants, due to similar surface charges of the humic acids and wood cells.

Table 1. Freundlich and Langmuir isotherm parameters for adsorption of organic compounds onto PP/PE fibres and pine bark.

	PP/PE fibres			Pine bark		
	Freundlich model parameters					
	R^2	n	K_F	R^2	n	K_F
FL	0.997	0.90	1.5	0.995	1.0	2.7
ANT	0.987	0.78	2.0	0.851	1.2	3.2
PYR	0.935	0.66	2.0	0.947	1.1	2.9
OP	0.980	0.79	0.27	0.984	1.0	2.8
NP	0.993	0.84	0.57	0.992	1.0	2.7
DBP	0.971	0.72	0.4	0.983	1.0	2.8
DEHP	0.345	0.49	2.7	0.795	1.3	3.5
	Langmuir model parameters					
	R^2	q_{max}	K_L	R^2	q_{max}	K_L
FL	0.997	-45	$-4.7 \cdot 10^{-2}$	1.000	-75	$-2.1 \cdot 10^{-2}$
ANT	0.998	-24	-0.15	0.969	-24	-0.13
PYR	0.997	-15	-0.21	0.994	2500	$1.2 \cdot 10^{-3}$
OP	1.000	-19	$-4.6 \cdot 10^{-3}$	1.000	-42	$-1.9 \cdot 10^{-2}$
NP	1.000	-37	$-9.6 \cdot 10^{-3}$	0.929	-2.0	$-7.6 \cdot 10^{-2}$
DBP	0.999	-18	$-1.2 \cdot 10^{-2}$	0.999	-14	$-2.9 \cdot 10^{-3}$
DEHP	0.162	-3.7	$-5.0 \cdot 10^{-2}$	0.943	46	$3.1 \cdot 10^{-3}$

The PP/PE fibres and pine bark showed linear adsorption capacity in the concentration range tested (Figure 1b). Higher concentrations could not be tested due to the analytes' limited water solubility. Pine bark and PP/PE fibres showed similar adsorption capacities for PAHs: approximately 1.4–44 μg/g, the highest capacity corresponding to the highest spike level (Figure 1b). Pine bark demonstrated the highest adsorption capacities for alkylphenols (1.3–41 and 0.9–42 μg/g for OP and NP, respectively) and DBP (1.1–41 μg/g). The PP/PE fibres adsorbed DEHP to the highest degree (0.6–42 μg/g), while its adsorption capacity for OP (0.6–27μg/g), NP (1.1–37μg/g) and DBP (0.9–37μg/g) were lower than for bark. However, no significant difference in adsorption capacity was found between the PP/PE fibres and pine bark (independent samples *t*-test, p>.005 for all spike levels), indicating that no material was generally better able to adsorb all tested compounds. Boving and Zhang (2004) presented similar capacities for adsorption of FL, ANT and PYR onto aspen wood (up to 12 μg/g, maximum initial concentration 50 μg/L). Similarly, adsorption capacities up to 70 μg/g for FL and benzyl butyl phthalate were reported by Ray *et al.* (2006) for hardwood mulch (maximum initial concentration ~400 μg/L). As a comparison, adsorption capacities in the mg/g-range
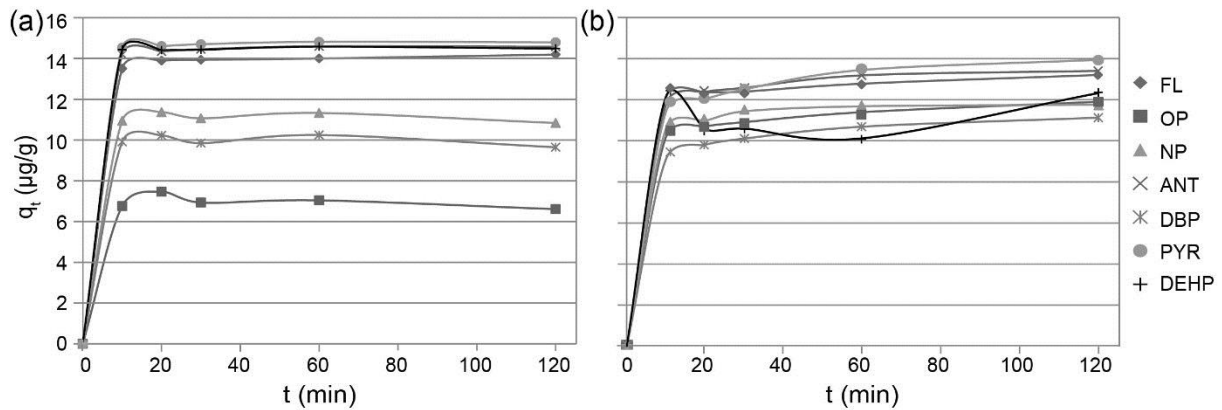
have been reported for PAHs, phthalates and alkylphenols on activated carbon (Julinová and Slavík 2012; Liu *et al.*2014; Yu *et al.* 2008).

Based on the correlation coefficients for the linearized plots of model equations (Table 1), the Langmuir adsorption model wasbetter able than the Freundlich model to describe experimental data for most compounds' sorption onto PP/PE fibres and bark. However, most compounds exhibited negative q_{max}:s, indicating that the Langmuir model cannot adequately explain the adsorption process. In addition, adsorption of DEHP onto PP/PE fibres could not be predicted well by any of the models. The Langmuir model assumes monolayer molecular adsorption, while the Freundlich model describes equilibrium on heterogeneous surfaces where monolayer and multilayer adsorption may occur (Boparai *et al.* 2011). Monolayer adsorption means that once a site is filled, no further sorption can take place and saturation is achieved. Since the Langmuir model assumes monolayer adsorption, it is said to better describe chemisorption than physisorption (Ruthven 2008). Chemisorption implies covalent, ionic and metallic bonds that are highly specific, whereas physisorption is attributed to van der Waals forces. However, because of the negative q_{max}:s for the Langmuir model, high adsorption capacity of PP/PE fibres and pine bark, and the many possible sorption interactions among sorbents and sorbates, the Langmuir isotherm is not appropriate, and physisorption should not be ruled out. In addition, it have been suggested (Boparai *et al.* 2011)that Freundlich *n*>1 indicate chemisorption. Considering that all Freundlich *n*<1 for PP/PE fibres (Table 1), we feel that physisorption and multilayer coverage is possible for this material.

The PP and PE are non-polar polymers; adsorption of the non-polar PAHs is therefore more likely to be higher than for the alkylphenol and phthalates (Figure 1a), which have both hydrophobic hydrocarbon chains and hydrophilic hydroxyl and ester groups, respectively. Both the non-polar PAHs and the polar alkylphenols and phthalates may be attracted to wood-based media as wood cells contain both non-polar parts, for example methyl groups, which may adsorb hydrophobic compounds, and oxygen-containing groups, including carboxyl (COOH), hydroxyl (OH) and carbonyl (C=O) groups, which provide adsorption sites for polar compounds (Rowell *et al.* 2012). Although only slight differences in adsorption capacities are seen between PAHs and other tested compounds on pine bark, the lower adsorption of alkylphenols and phthalates may be due to competition from both non-polar and polar molecules, including humic acids, water and other organic pollutants. In addition, the alkylphenols and phthalates, especially DEHP and OP, are bulkier than the PAHs and internal pores of the media surfaces may be inaccessible to these molecules.

Kinetics
Adsorption is a time-dependent process, and it is necessary to evaluate an optimal contact time between the organic compounds and the adsorbents for design and use, e.g. in filters. Cellulose and zeolite was not included in the kinetic tests due to their low adsorption capacities. The adsorption of compounds occurred within 10 min in contact with PP/PE fibres (Figure 2a). For pine bark, maximum sorption was not attained after 120 min(Figure 2b), but the differences in removal at 30 min and 24 h (70–85%, Figure 1a) were generally onlyaround 10%. A similar adsorption equilibrium process has been reported for other plant-based media, such as metals on mulch (Jang *et al.* 2005) and sawdust (Bulut and Tez 2007), and bisphenol A onto natural sorbents such as rice husk, sawdust and peat (Zhou *et al.* 2012).

Figure 2. Adsorption capacities (q_t, μg/g) of PP/PE fibres (a) and pine bark (b) as a function of time (t, min).

Table 2. Correlation coefficients (R^2) for first- and second-order linear model plots, and second-order model parameters for adsorption on PP/PE fibres and pine bark.

	First order	Second order			
		PP/PE fibres			
	R^2	R^2	k_2	q_e, calc	q_e, exp
FL	0.178	1.000	0.106	14.25	14.18
ANT	0.450	1.000	0.230	14.62	14.60
PYR	n/a	1.000	0.308	14.81	14.58
OP	n/a	0.999	0.083	6.56	6.15
NP	0.016	0.999	0.102	10.82	11.11
DBP	0.131	0.999	0.088	9.62	10.44
DEHP	n/a	1.000	1.582	14.51	14.24
		Pine bark			
FL	0.996	1.000	0.042	13.33	13.82
ANT	0.886	1.000	0.037	13.61	14.26
PYR	0.988	1.000	0.020	14.31	14.23
OP	0.969	1.000	0.030	12.12	13.04
NP	0.700	1.000	0.064	11.89	13.50
DBP	0.918	1.000	0.027	11.39	12.58
DEHP	0.023	0.986	0.018	12.36	10.42

n/a, not applicable, due to $q_e=q_t$ for some time points, its log could not be calculated and plotted.

Similar to results presented by such authors as Bulut and Tez (2007) and Jang *et al.* (2005) for metal adsorption onto sawdust and mulch, respectively, plotting the first-order kinetic model for pine bark indicates that this equation is only valid for certain compounds during the initial 0–30 min of the adsorption process. Even weaker agreementwith the first-order equation wasfound for PP/PE fibres (Table 2). However, for the second-order kinetic model, $R^2 \geq 0.99$for all compounds(Table 2), indicating that the pseudo-second-order model fits the adsorption process well in this study. The experimental and calculated q_e:s for all compounds are in good agreement (Table 2), which further supportsthe pseudo-second-order model forthe adsorption kinetics in this study. The rate constants for PP/PE fibres ($M = 0.41$, $SD = 0.53$)are not significantly higher than for pine bark ($M = 0.05$, $SD = 0.04$), $t(14) = 1.93$, $p = .095$.

CONCLUSIONS

- Adsorption tests show that fibres of PP/PE and pine bark have great potential for use in filters for stormwater. These materials are inexpensive and abundant, show high sorption capacity (up to 44 µg/g) and fast removal (>70% within 30 min) of organic pollutants frequently present in stormwater.
- The capacity of cellulose and zeolite to adsorb organic pollutants was negligible. However, these materials may serve as efficient adsorbents of metals in stormwater because of their potential to attract cations.

It is our recommendation that further research investigateswhether the filter materials areeffective inremoving pollutants from naturalstormwater, where many influencing factors, such as fluctuating pollutant concentrations, water flow, particle content and colloidal transport, can affect the adsorptioncapacity of the material.

ACKNOWLEDGEMENTS

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