

Removal of selected pollutants from aqueous media by hardwood mulch

Asim B. Ray*, Ariamalar Selvakumar, Anthony N. Tafuri

Urban Watershed Management Branch (MS-104), United States Environmental Protection Agency, 2890 Woodbridge Avenue, Edison, NJ 08837, USA

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Abstract

Generic hardwood mulch, usually used for landscaping, was utilized to remove several selected pollutants (heavy metals and toxic organic compounds) typically found in urban stormwater (SW) runoff. The hardwood mulch sorbed all the selected pollutants from a spiked stormwater mixture, including copper (Cu^{2+}), cadmium (Cd^{2+}), chromium (Cr^{6+}), lead (Pb^{2+}), zinc (Zn^{2+}), 1,3 dichlorobenzene (DCB), naphthalene (NP), fluoranthene (FA), butylbenzylphthalate (BBP), and benzo(a)pyrene (B[a]P). Masses of the pollutants sorbed depended upon the pollutant species, contact time, and initial concentration which varied from 20 to 100%. Sorption rates of the metals, in general, were more rapid than those of the organics; however, mass removals (percent) of the organics, in contrast to those of the metals, were independent of their initial concentrations. With the exception of Cd, percentages (weight) of the metals removed declined as their initial concentrations decreased. None of the sorbed pollutants desorbed to any significant extent upon extended washing with water. It is quite feasible that in the presence of mulch the uptake of these pollutants by the aquatic species will be reduced significantly.

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1. Introduction

Pollutants found in urban stormwater (SW) runoff originate from a number of sources: washings of road surfaces, parking lots, motor vehicles, building materials, air pollutants, exhausts from industrial operations, and lawn fertilizers. The heavy metals of most concern in urban stormwater runoff are lead, zinc, copper, chromium, cadmium, and nickel [1]. Like heavy metals, toxic organic compounds such as polycyclic aromatic hydrocarbons (PAHs) are found in higher concentrations in urban stormwater runoff than in rural areas. For example, some of the most prevalent PAHs like phenanthrene, fluoranthene (FA), pyrene, and benzopyrenes were found at higher concentrations in stormwater runoff from the city of Columbia, North Carolina, than in rural areas [2]. Both the species and concentrations of the pollutants in the runoff, however, depend on a number of factors including intensity and duration of the storms, length of the antecedent dry period, effectiveness of waste removal from streets, land use, etc.

Stormwater runoff, because of its heavy metals and PAHs content, is considered toxic and unacceptable for discharge into receiving surface waters without some kind of treatment [1]. Typically, a number of devices, collectively known as structural Best Management Practices (BMPs), are employed to treat these contaminated runoffs. Since, the majority of these structural BMPs (e.g., wet ponds) rely exclusively on physical separation processes such as sedimentation only a majority of the suspended solids and associated pollutants are removed without any significant decrease in soluble pollutant concentrations. For instance, Boving [3] in his study of the removal efficiency of a newly constructed detention pond in Providence, RI, treating highway runoff, observed no significant change in the concentrations of dissolved PAHs entering and leaving the pond.

Physical processes such as activated carbon adsorption (mostly for organic compounds), or ionic exchange with synthetic resins (usually for metals) are typically used to remove trace quantities of toxic compounds from water [4]. These processes, although very effective, are rather expensive because of the materials involved. Consequently, the possibility of using alternative materials easily available at a low cost were explored [5]. A number of natural materials such as pine bark [6], tree fern [7], rice husk [8], and wood fibers [9] have been used to remove dissolved pollutants from aqueous media. For practical

* Corresponding author. Tel.: +1 732 906 6822; fax: +1 732 906 6896.
E-mail address: ray.asim@epa.gov (A.B. Ray).

purposes, to improve solid–liquid contact and to prevent channeling, these natural media are often mixed with sand when used in filtering contaminated stormwater runoff [10].

Most of the published studies evaluating the performance of natural organic filter media are limited to either toxic organic compounds or heavy metals but not a mixture of both pollutant species. In this study, a generic hardwood mulch was used to treat a synthetic stormwater runoff spiked at pre-determined concentrations with 10 pre-selected priority pollutants including the heavy metals: copper (Cu^{2+}), cadmium (Cd^{2+}), hexavalent chromium (Cr^{6+}), lead (Pb^{2+}), and zinc (Zn^{2+}); and the toxic organics naphthalene (NP), fluoranthene, 1,3 dichlorobenzene (DCB), benzo[a]pyrene (BaP), and butylbenzylphthalate (BBP).

The objectives of this study are to:

1. determine the feasibility of using a generic hardwood mulch, as a filter medium, to remove soluble pollutants, both heavy metals and toxic organic compounds, commonly found in urban stormwater runoff;
2. determine the sorption characteristics of the mulch as a function of the concentrations of the pollutants and the stability of the sorbed pollutants towards weathering, rain, for example;
3. develop a correlation between the sorption capacity of the mulch and a physical property of the pollutants; and
4. determine the influence of the native mulch pollutants on the outcomes of the study.

2. Experimental design

2.1. Materials

Synthetic stormwater runoff (stock solution) was prepared by dissolving analytical grade potassium dichromate, metal nitrates and the pre-selected organic compounds in deionized water. Concentrations of the pollutants in the stock solution were determined by inductively coupled plasma spectroscopy (metals) and gas chromatography/mass spectroscopy (organics) (Table 1).

Hardwood mulch was obtained from a local (New Jersey) garden store. This commercial landscaping mulch was a mixture of various sizes (Table 2) of Silver Maple, Norway Maple, Red Oak, and Cherry and appeared to be coated with mineral oil (possibly from the tree shredding operation). Soil particles

Table 1
Concentrations of pollutants in stock solution (pH 4.60)

Metals	Concentrations (mg/L)	Organics	Concentrations ($\mu\text{g/L}$)
Copper (Cu)	0.992	Dichlorobenzene (DCB)	47
Cadmium (Cd)	1.016	Naphthalene (NP)	375
Chromium (Cr^{+6})	0.989	Fluoranthene (FA)	418
Lead (Pb)	0.996	Butylbenzylphthalate (BBP)	400
Zinc (Zn)	1.079	Benzo(a)pyrene (B[a]P)	851

Table 2
Particle size distribution of mulch (dried at 40°C)

Mesh size	Opening size (μm)	% Retained
4	4760	36.5
8	2360	27.4
10	2000	4.6
20	850	20.5
40	20.5	6.8
Pan	<20.5	4.2

were also found adhering to the bulk mulch. The mulch was wet-screened with tap water through a # 4 mesh ($4760\ \mu\text{m}$). The ($-4760\ \mu\text{m}$) fraction was further agitated with tap water and again wet-screened to obtain the desired (-2360 to $+2000\ \mu\text{m}$) fraction used throughout the study. Wet screening of the landscaping mulch not only separated the desired fraction from the finer ones but also removed adhering soil particles and some mineral oil. The residual mineral oil, for this study, was considered inert.

2.1.1. Analytical methods and instrument

Analytical methods and instruments used in this study are summarized in Table 3.

All experiments were run in duplicate and the duplicates of the analytical data for each test were within $\pm 10\%$ of each other.

2.2. Characterization of mulch

The air-dried (40°C) mulch (-2360 to $+2000\ \mu\text{m}$ fraction) was analyzed for its elemental carbon, hydrogen, nitrogen, and sulfur composition, ash, and also for its ‘pollutants of interest’ content (Tables 4 and 5).

The feasibility of leaching of any of the indigenous ‘pollutants of interest’ (Table 5) from the mulch during the sorption

Table 3
Analytical methods and instruments

Measurement	Method	Instrumentation
Metals	a. Digestion: EPA 3015	ICP: PE OPTIMA 3000XL ICP-OES
a. Aqueous phase b. Solid phase	b. Digestion: EPA 3050 Analysis (a + b): EPA 6010	
Organics	a. Extraction: EPA 3510	GC/Mass: HP 5890/5971 GC-Mass
a. Aqueous phase b. Solid phase	b. Extraction: EPA 3540 (pyrene as matrix spike) Analysis (a + b): EPA 625	
C, H, N content	ASTM D 5373	CHN analyzer: PE 2400 CHN analyzer
S content	ASTM D 4239 (Method C)	Furnace with IR detection Leco CS-444
Moisture content	ASTM D 2974 (Method C)	Oven
Ash content	ASTM D 2974	Oven
Particle size distribution	ASTM D-828	Screen
pH	SM4500 – H ⁺ B	pH meter

Table 4
Characterization of mulch

% Moisture	% Ash		Elemental composition (dry basis)				
	550 °C	880 °C	% C	% H	% N	% S	% O ₂ (by diff)
60.30	11.30	10.90	44.12	5.86	0.63	0.16	38.33

studies was determined by agitating 20 g of the mulch, at room temperature, with 1 L of HCl (pH 4.0) for 1 h. The mulch–HCl mixture was vacuum filtered, the residue washed with 100 mL of distilled water, and filtered. The combined filtrates (1070 mL; pH 3.90) were analyzed for all the pre-selected pollutants (Table 5).

2.3. Sorption rate study

Twenty grams of air-dried mulch was added to each of the four beakers containing 2 L of stock solutions (Table 1). The watch-glass covered beakers were agitated with teflon-coated magnetic stirrers at the lowest speed at which the mulch remained in suspension. After 30, 60, 120, and 240 min of agitation, the contents of the beakers were vacuum filtered and the solids were washed with 100 mL of distilled water and then filtered. The volume, pH, and temperature of the combined filtrates were noted (Table 6). Dependence of contact time on percent of pollutants removed is shown in Table 7 and Figs. 1 and 2.

2.4. Equilibration of mulch with pollutant mixture of various concentrations

The stock solution (Table 1) was diluted (2-, 4-, and 8-fold) with distilled water to make up three additional stock solutions. Analyses of these three diluted solutions confirmed the concentrations of the pollutants in the three solutions were at 0.5, 0.25, and 0.125 fractions of their original concentrations, respectively. To each of these 3 L solutions (stock solution and the three diluted ones), in four cleaned (detergent, nitric acid, and distilled water) beakers, 25 g of air-dried mulch was added. The watch-glass covered beakers were agitated with teflon-coated magnetic stirrers for 72 h. Contents of the beakers were vacuum filtered, the solids washed with known volumes of distilled water, and volumes of the resulting liquids were recorded (Table 8). Both the solids and liquids were analyzed for the 10 pre-selected pol-

Table 5
Dissolution of indigenous mulch pollutants in HCl (pH 4.0)

Metals				Organics			
Concentration in mulch (mg/kg)	Concentration in filtrate after HCl treatment (mg/L)	% Leached		Concentration in mulch (μg/kg)	Concentration in filtrate after HCl treatment (μg/L)	% Leached	
Cu	4.32	ND	0.00	DCB	ND	NA	NA
Cd	ND	NA	NA	NP	114	ND	0.00
Cr	0.45	0.01	100	FA	220	ND	0.00
Pb	0.78	0.02	100	BBP	607	ND	0.00
Zn	13.90	0.07	27	B[a]P	ND	NA	NA

Note: Concentrations of all pollutants in the solids are on dry basis. ND, not detected. NA, not applicable.

Table 6
Volume, pH, and temperature of filtrates from sorption rate study

Contact time (h)	Combined filtrate volume (mL)	pH		Temperature (°C)
		Initial	Final	
0.5	2060	3.70	4.40	18.20
1.0	2065	3.65	4.45	18.00
2.0	2065	3.60	4.60	18.00
4.0	2060	3.60	4.75	19.50
72.0	2060	3.70	4.80	19.50

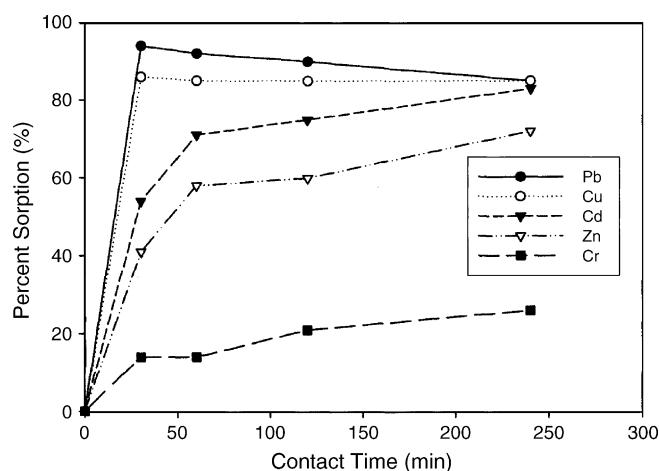


Fig. 1. Percent sorption vs. contact time for metals.

lutants. The pH of the mixtures, before and after filtration, were also noted. Percentages of the pollutants sorbed as a function of their initial concentrations are shown in Table 9 and Figs. 3 and 4.

2.5. Desorption studies

To determine the stabilities of the sorbed pollutants to weathering (e.g., rain) 15 g of mulch, equilibrated with the stock solution (Table 1) for 72 h (Table 5), was agitated with 1.5 L of distilled water. The pH of the mixture was adjusted to 6.0 and stirred for 1 h with a teflon-coated magnetic stirrer. The resulting mixture was vacuum filtered. Volume and pH of the filtrate were noted and analyzed for all the pre-selected pollutants (Table 10).

Table 7
Sorption capacity of hardwood mulch vs. contact time

Pollutant	Physical property	Contact time (h)				
		0.5	1.0	2.0	4.0	72
Pollutant { % removal }, concentration ($\mu\text{mole/g}$), and concentration [$\mu\text{g/g}$]						
Cu ²⁺	0.72 ^a	{86} (2.52) [160]	{86} (2.52) [160]	{85} (2.50) [158]	{85} (2.50) [158]	{85} (2.50) [158]
Cd ²⁺	0.97 ^a	{54} (0.90) [100]	{71} (1.20) [135]	{75} (1.27) [143]	{83} (1.41) [158]	{86} (1.46) [164]
Cr ⁶⁺ /Cr ³⁺	0.52 ^a /0.63 ^a	{14} (0.50) [26]	{14} (0.50) [26]	{21} (0.75) [39]	{26} (0.93) [48]	{68} (2.43) [126]
Pb ²⁺	1.20 ^a	{94} (0.85) [176]	{92} (0.85) [172]	{90} (0.81) [168]	{85} (0.77) [159]	{92} (0.83) [172]
Zn ²⁺	0.74 ^a	{41} (1.27) [83]	{58} (1.80) [117]	{60} (1.86) [121]	{72} (2.23) [146]	{72} (2.23) [146]
DCB	147 ^b 113.1 ^c	{4} (0.002) [0.30]	{64} (0.04) [5.70]	{63} (0.03) [5.10]	{71} (0.04) [6.10]	{100} (0.08) [12.34]
NP	128 ^b 148 ^c	{15} (0.08) [10.55]	{61} (0.34) [42.92]	{63} (0.35) [44.32]	{65} (0.36) [45.80]	{88} (0.48) [61.91]
FA	202 ^b 217 ^c	{82} (0.32) [64.30]	{89} (0.35) [69.80]	{89} (0.35) [69.80]	{95} (0.37) [74.50]	{93} (0.36) [72.90]
BBP	312 ^b 278.7 ^c	{28} (0.07) [21]	{90} (0.22) [67.54]	{90} (0.22) [67.54]	{95} (0.23) [71.30]	{77} (0.19) [57.80]
B[a]P	252 ^b 263 ^c	{59} (0.37) [94.20]	{80} (0.51) [127.7]	{80} (0.51) [127.7]	{84} (0.53) [134.00]	{92} (0.58) [146.90]

^a Ionic radius (\AA).

^b Molecular weight (g).

^c Molecular volume (cm^3).

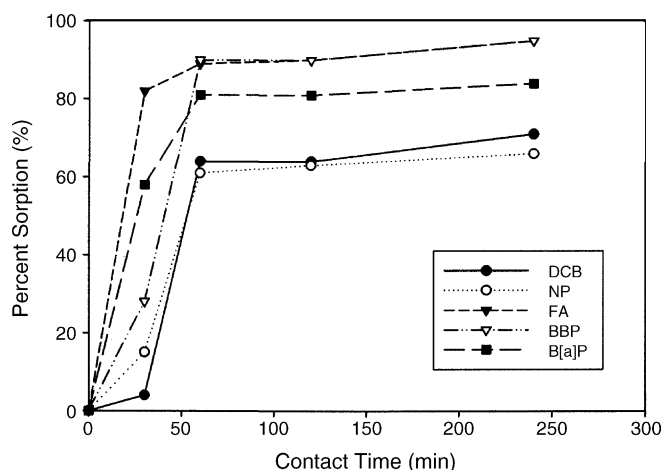


Fig. 2. Percent sorption vs. contact time for organics.

Table 8
Volume, pH, and temperature of filtrates from equilibrium study

Pollutant concentration ($\mu\text{g/L}$)	Total volume (mL)	pH		Temperature ($^{\circ}\text{C}$)
		Initial	Final	
~1000	3070	3.80	4.95	19.50
~500	3080	4.10	5.90	23.80
~250	3080	4.80	6.40	24.20
~125	3080	4.80	6.90	24.00

Table 9
Percent removal vs. initial concentration of pollutants

Pollutants	Initial concentrations ($\mu\text{g/L}$) and removal [%]			
Cu	(992) [87]	(496) [77]	(248) [59]	(124) [0]
Cd	(1016) [86]	(508) [98]	(254) [100]	(127) [100]
Cr ⁶⁺	(989) [68]	(495) [42]	(247) [0]	(124) [0]
Pb	(996) [92]	(498) [92]	(249) [87]	(125) [84]
Zn	(1079) [72]	(540) [81]	(270) [73]	(135) [43]
DCB	(47) [100]	(24) [100]	(12) [100]	(6) [100]
NP	(375) [88]	(188) [82]	(94) [100]	(47) [100]
FA	(418) [93]	(209) [90]	(105) [91]	(53) [100]
BBP	(400) [77]	(200) [100]	(100) [100]	(50) [100]
B[a]P	(851) [92]	(426) [90]	(213) [90]	(1070) [97]

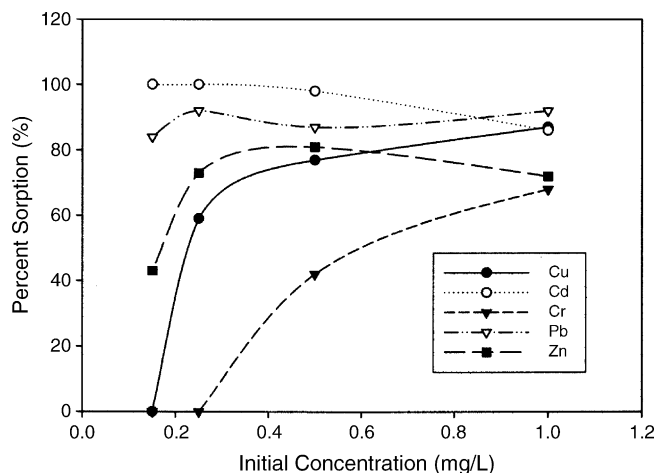


Fig. 3. Percent sorption vs. initial concentration for metals.

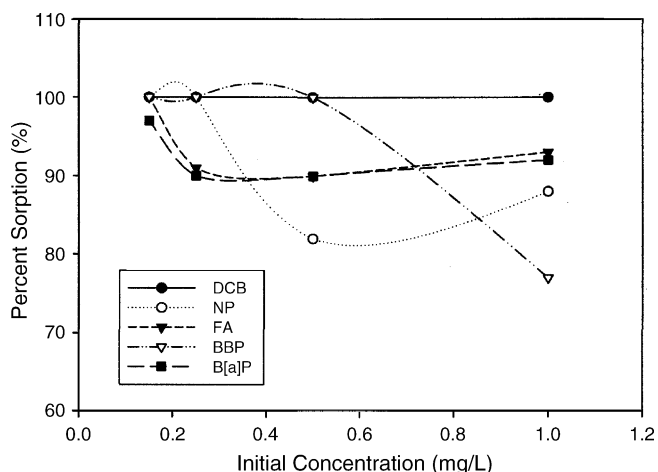


Fig. 4. Percent sorption vs. initial concentration for organics.

Table 10
Desorption of sorbed pollutants on washing

	Heavy metals concn, in mulch ($\mu\text{g/g}$)		% Desorbed		Toxic organics concn, in mulch ($\mu\text{g/g}$)		% Desorbed
	Before desorption	After desorption			Before desorption	After desorption	
Cu	24.61	24.14	1.90	DCB	1.92	1.92	0.00
Cd	25.55	25.55	0.00	NP	9.19	8.72	5.10
Cr	19.63	17.45	11.10	FA	11.37	10.75	5.45
Pb	26.79	26.01	2.90	BBP	9.03	9.03	0.00
Zn	22.74	20.56	9.60	B[a]P	22.90	22.90	0.00

Note: All results on dry basis.

3. Results and discussion

3.1. Characterization of mulch

The wet-screened mulch fraction, with the exception of Cd, DCB, and benzo(a)pyrene (B[a]P), contained the other seven “pollutants of interest” in small but significant concentrations (Table 5). Dilute HCl treatment (pH 4.0) of the screened mulch showed different leaching characteristics of the pollutant species. Although none of the indigenous organic compounds of the mulch was affected by the acid treatment, most of the native metals dissolved in dissimilar quantities; e.g., Cu was unaffected, 27% of the Zn present dissolved, and both Cr and Pb leached completely (Table 5). Because of the higher pH conditions (pH \approx 6.0) of this study (Table 8), it is very likely that smaller quantities of metals will dissolve from the mulch. As the actual concentrations of the metals leached compared to those in the stock solution (e.g., 0.01 mg/L versus 0.998 mg/L for Cr, 0.02 mg/L versus 0.996 mg/L for Pb, and 0.07 mg/L versus 1.08 mg/L for Zn) and the most diluted one employed (Table 10) (e.g., 124 $\mu\text{g/L}$ versus 10 $\mu\text{g/L}$ for Cr, 125 $\mu\text{g/L}$ versus 20 $\mu\text{g/L}$ for Pb, and 135 $\mu\text{g/L}$ versus 70 $\mu\text{g/L}$ for Zn), the effects of the indigenous mulch metals, with the exception of Zn, on the results of the study are not expected to be very significant.

3.2. Sorption rate study

Under the conditions of this study, the removal rates (measured as % removed) of the metals, with the exception of Cr^{6+} , were quite rapid (Fig. 1). Sorption rates of the metals throughout the experimental period persisted in the following order $\text{Pb} \geq \text{Cu} > \text{Cd} > \text{Zn} > \text{Cr}^{6+}$ (Fig. 1). The comparatively sluggish removal rate and low removal (%) of Cr^{6+} can be ascribed to the very nature of the Cr^{6+} sorption process. The actual adsorption process for Cr^{6+} , unlike other metals, consists of two separate reaction steps; i.e., reduction of Cr^{6+} to Cr^{3+} by the mulch, followed by sorption of the Cr^{3+} produced. The sorption rate, therefore, will depend upon the rate of reduction of Cr^{6+} , a comparatively slow process [11]. Consequently, the amount of Cr^{6+} removed will depend upon the quantity of Cr^{3+} produced. The validity of this assumption is corroborated by the findings that upon increasing the contact time from 4 to 72 h (Table 7), more Cr^{6+} (68% versus 26%) was removed from solution.

Profiles of the sorption rate curves of the organics (Fig. 2) are comparable to those of the metals (Fig. 1). Based on percent

(weight) removal, the sorption rates of these compounds were in the order: $\text{B[a]P} > \text{FA} > \text{NP} > \text{BBP} > \text{DCB}$. However, compared to the metals (excluding Cr^{6+}) the sorption rates of these organic compounds were slow.

A review of the sorption capacity of the mulch (72 h of contact time, Table 7) for the metals and the organics showed that the sorption capacity of the mulch can be correlated with their ionic radii and molecular weights/volume, respectively. For example, the sorption capacity of the mulch for the five metals (including Cr^{6+}) can be correlated (Fig. 1) by the equation:

$$y = -2.63x + 4.07 \quad (r^2 = 0.95)$$

where y is the moles of metal sorbed/g of dry mulch; and x is the ionic radius of the metal in \AA .

The degree of correlation between these two quantities seems to improve when only the metals with the same electrical charge (valence) are considered. For example, eliminating both Cr^{3+} and Cr^{6+} the correlation becomes:

$$y = -3.33x + 4.51 \quad (r^2 = 0.99)$$

Relationship between molecular weight or volume of the PAHs and their percent removal, in line with the earlier observations [12], with $r^2 = 0.68$ and 0.74 , respectively, was also observed in this study (Table 7). The correlation coefficient, however, like the metals, becomes better ($r^2 = 0.84$) when only the organic compounds of the same type, such as planar and non-polar NP, FA, and B[a]P molecules are considered. Attempts to correlate absolute mass-removal efficiency ($\mu\text{g/g}$) of these organics with the molecular weight or hydrophobicity [9] were not successful. However, better correlation was obtained when only compounds with similar characteristics, PAHs without any substituents, for example, are considered.

3.3. Equilibration of mulch with pollutant mixtures at several concentrations

The objective of these tests was to determine the sorption characteristics of the mulch as a function of the concentrations of the pollutants. The results (Table 9) show that the sorption capability, measured as percent removal, of the mulch varies not only with the changes in the concentrations of the pollutants but also with their species. For example, while lowering of the initial concentrations hardly affected the removal capacity (percent removal) for the organics; however, it had adverse effects on metals removal. Of the metals, only Cd, and to a certain extent Pb,

were not affected but the ability of the mulch to remove Cu, Cr⁶⁺, and Zn, at lower concentrations were reduced severely (Table 9 and Fig. 3). Concentrations at which these declines occurred, however, varied also with the metal species. For example, for Cu this abrupt change occurred at a concentration of 124 µg/L (1.95 µmole/L) while for Cr⁶⁺ and Zn this decline ensued at significantly higher concentrations, 247 µg/L (4.75 µmole/L) and 135 µg/L (2.06 µmole/L), respectively.

Sorption capability, determined as percent removal, of the mulch for the PAHs, in contrast to the metals, did not decline with the reduction in their initial concentrations but increased as the ratio (weight) of the sorbent to the pollutants rose. The most noticeable feature of the sorption of the organics by the hardwood mulch is its ability to remove very low concentrations of toxic organic compounds from aqueous mixtures of heavy metals and toxic organic compounds. For example, 100% of DCB was removed from an aqueous mixture of the 10 pollutants containing only 6 ppb of DCB (Table 9 and Fig. 4). It is noteworthy that the pH of the initial pollutant mixture changed from 4.8 to ~7.0 on treatment with the mulch, an indication of its potential acid neutralization capability.

3.4. Desorption study

Amounts of pollutants desorbed on washing the mulch with deionized water varied (Table 10). The loss, however, in no case exceeded more than 10% of the sorbed amount, with the exception of Cr where the loss on washing was more than 10%. Based on loss on washing, the binding strength of the metal to mulch appears to be in the order: Cd > Cu > Pb > Zn > Cr. Losses of the sorbed heavy metals on washing are probably due to two different types of sorption, chemical versus physical, of the metals to humic materials [13] of the mulch. These sorbed toxic organics seemed to be held more strongly (Table 10) than the heavy metals studied. Based on the results of desorption on washing the stability of the bond between the organic compounds and the mulch appears to be in the order: B[a]P ≥ BBP ≥ DCB ≥ FA > NA.

4. Conclusion

The results of this study show that common garden store variety hardwood mulch can be used to remove water soluble pollutants such as heavy metals and toxic organic compounds normally found in stormwater runoff. These strongly held sorbed pollutants are very resistant to further washing with water. This observation not only tends to support the previous findings [12] that in the presence of soot and soot-like materials 'uptake of

toxic organic compounds like polycyclic aromatic hydrocarbons and polychlorinated biphenyls by aquatic organisms can be reduced significantly' [12] but hardwood mulch can be used to reduce the uptake of heavy metals by the same organisms as well.

Results of hydrochloric acid leaching of the mulch (Table 5) along with the above observations, raises the intriguing possibility that on ageing, desorption of the sorbed pollutants can be reduced further to the point that the usual disposal/toxicity problems associated with these materials can be reduced substantially.

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