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Treatment of wood leachate with high polyphenols content by peat and carbon-containing fly ash filters

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ABSTRACT

In the present study, two combinations of filter materials in filter/columns were examined for removal of total organic carbon (TOC) and polyphenols (PP) found in storm water runoff from wood storage areas in a wooden floor industry. One filter/column was packed with peat mixed with carbon-containing fly ash, while another filter/column contained only peat (without ash). The mixture of peat and ash has shown faster and higher removal capacity for TOC and faster removal with the same final removal capacity for PP (in grams of pollutant per kg of sorbent) at the saturation point. The superiority observed for the peat and ash filter is presumably due to the unique characteristics of peat and ash, which enhanced the treatment efficiency when used together in a mixture. Based on the observed results, filters formed by peat and carbon-containing ashes proved to be a potentially low-cost option for the treatment of storm water generated at storage areas of wood materials such as logs, sawdust and wood chips.

Keywords: Fly ash; Peat; Adsorption; Polyphenols; TOC; Industrial storm water; Wood residues

1. Introduction

Wood industries generate several types of wastewaters including storm water, process wastewater and irrigation wastewater, depending on the indoor and outdoor activities. Due to the large storage areas with logs, bark or sawdust and mainly after rainfall, snow melt and irrigation of logs, most wood-based industries generate polluted storm water runoff because of leaching of organic and inorganic compounds from wood materials. The literature refers to storm water runoff from wood-based industries [1] and storage areas of wood waste in landfills [2] as potential hazardous for receiving water bodies. The expanding bio-energy industry with large outdoor wood storage areas at power plants, particularly in Europe has

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given rise to increasing volumes of wood leachate. Furthermore, veneer application generates a process water (boiling water) which is usually highly contaminated with wood leachate [3], with similar properties to that of storm water from log yards and therefore, it is also a potential candidate for treatment.

Wood leachate is characterized by the high content of organic carbon [1]. It has low pH and is highly coloured, probably due to humic acids and different polyphenols (PP) such as tannic acids or lignin [4]. Wood leachate is also often reported as toxic to aquatic organisms [1] such as *Daphnia magna* [5], *Artemia salina* [6] and *Oncorhynchus mykiss* (rainbow trout) [4]. Many different compounds in wood leachate are described as potentially toxic such as tannins, lignin, tropolone, terpene, lignans as well as have the low pH [7]. Moreover, different tree species generate leachate with different compositions [3,6]. The leachate is generated when the water comes in contact with wood and the resulting leachate usually has intense colour, high tannins content (measured as total PP) and cause toxic effects to aquatic organisms [6].

Different methods for wood leachate treatment have been described in the literature; and some of these methods are space dependent such as constructed wetlands [8] or planted soil infiltration systems [9]. Less space-depending methods include ozonation [10] and reverse osmosis [11] which require proper equipment and more complex procedures.

The present paper reports the treatability study using sorption materials as filters for storm water runoff. The aim was to compare the sorption materials in treating leachate pollutants with focus on total organic carbon (TOC) and PP. A mixed filter with peat and carbon-containing fly ash and only peat were selected. Peat and ash were previously applied for the treatment of industrial landfill leachate [12] and wastewater from car washing [13]. These inexpensive sorption materials are naturally occurring or by-products of some industrial manufacturing process and they are also considered to be an environmentally friendly option. Since these filter materials have been proved to reduce the phenols concentrations in leachate from landfills, it was assumed that it should be a good candidate to reduce PP content in the leachate generated at wood storage areas.

2. Materials and methods

2.1. Storm water runoff

Storm water runoff contaminated with leachate from pedunculate oak (*Quercus robur*) storage area was collected and stored in 1 m^3 tanks at room temperature. The storm water was collected from a ditch directly fed with runoff from a large wood chip storage pile mainly consisted of oak wood chips in a wooden floor industry. To ensure the quality of the storm water, the chemical composition was regularly checked during four months with sampling every second week for the analysis of TOC, pH, electrical conductivity (EC), colour and PP using established methods (see chemical analyses).

2.2. Filter material

The filter material used was a mixture of peat and carbon-containing fly ash [14] with a ratio of 3:1 (v/v). This filter containing a mixture of peat and ash has been previously proved to reduce metals and organic pollutants from the water phase; it has also been found that it contains phenol-degrading bacteria [14–16]. The material used for the filter was acquired from a supplying company (Laqua Treatment AB). Both raw peat and peat mixed with carbon-containing ash to a ratio of 3:1 (v/v) was used as previously described [16]. The dry mass content of the filter material was measured by drying at 100˚C during 24 h.

2.2.1. In situ *porosity of the filter bed*

The *in situ* porosity was determined by immersing the filter bed in water [15]. The total volume of voids is represented by the total amount of water within the filter, which is estimated by the water that is released by gravity plus the water trapped in the filter material after leaking stops, which is also measured as the difference in weight before and after drying the filter material.

2.3. Experimental set-up

In total, 12 columns of PVC with an inner diameter of 70 mm and height of 1 m were filled with the filter material; six columns were filled with peat and another six with mixed peat and ash (Fig. 1) in order to examine the sorption capacity of the two materials under dynamic conditions. Uncertainties, such as time for the breakthrough to occur and the risk of facing lack of storm water to feed the system were solved by packing six columns up to a level of 0.8 m and six columns up to a level of 0.4 m. The total volume of the columns with 0.8 m filter material was 3 L, meanwhile the total volume of the columns with 0.4 m filter material was 1.5 L. The density of the peat was about 140 g L^{-1}, whereas the density of the mixture (peat and ash) was about 280 g L^{-1}. The leachate was pumped onto the columns (downwards flow mode) for 60 min twice a day at a flow rate of 9 mL min^{-1}

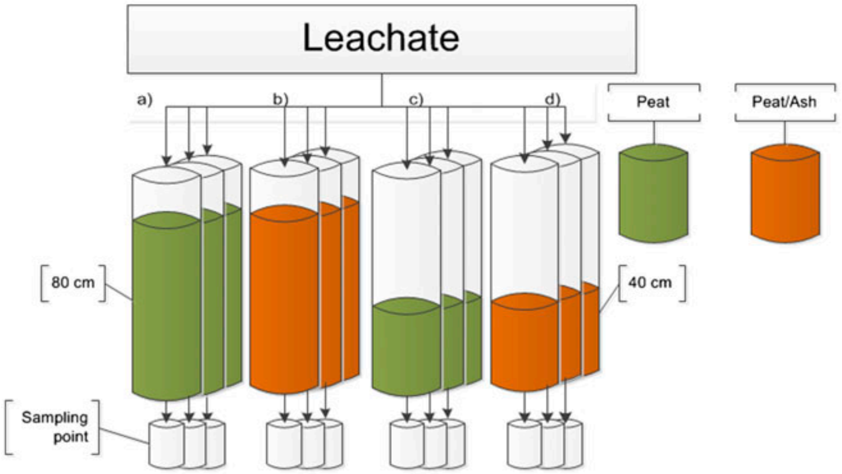

Fig. 1. Four experimental set-ups: columns filled with up to 80 cm (a and b) and; 40 cm (c and d) of only peat (a and c) and; peat + ash (b and d).

(1.1 L d^{-1}). This flow was previously applied for leachate treatment [16] and it was selected in order to mimic as much as possible full-scale conditions, where the flow depends on the amount of storm water to be treated.

It is known that up-flow mode maximizes the retention time. However, downward flow is the easiest way to construct and operate a full-scale system, since it is difficult to construct an up-flow system using material that floats easily such as peat and fly ash [16].

During 125 days of operation, 136 L of wood leachate was pumped through each column. Samples were collected every 14th day with a total of 8–10 samples collected from each column. The results were calculated as gram of pollutant reduced per kg of filter. This way of reporting (pollutant mass reduced per filter mass) makes it possible to estimate the amount of filter material needed to reduce a certain amount of any targeted pollutant, keeping in mind that real wastewaters might contain a number of pollutants which might also be removed by the filter.

2.4. Chemical analyses

The pH was measured with a Mettler Toledo SG2. EC was measured with Hach HQ40D. PP were quantified using Folin–Ciocalteu reagent [17] and reported as total content in mg L^{-1} tannic acid equivalent. Chemical oxygen demand (COD) was analysed spectrophotometrically by Dr 5000 with Dr Lange test kits LCK 114 (Hach-Lange, Dusseldorf). TOC was analysed spectrophotometrically by Dr 5000 with Dr Lange test kits (LCK 386 and LCK 387) (Hach-Lange, Dusseldorf). The TOC measurement is made in a two-stage process. The total inorganic carbon is first eliminated with the help of a shaker and then the TOC is oxidized with persulphate to carbon dioxide (CO_2). The CO_2 passes through a membrane into the indicator cuvette, where it causes colour change, which is evaluated with a Dr Lange 5000 photometer. Colour was measured by Hach spectrophotometer Dr Lange 5000 according to the method 8025 and expressed as mg L^{-1} Pt-Co.

2.5. Fourier transform infrared (FTIR) spectroscopy

FTIR spectroscopy spectra were recorded at room temperature (about 22°C) using a FTIR spectrometer (Avatar system 320) equipped with Ez Omnic 6.2 software. FTIR spectra of different samples were obtained. The sample preparation was performed by grinding solid samples before mixing them with KBr (FTIR grade, Fluka) previously dried at 45°C for 24 h. CO_2, aqueous vapor in air and possible KBr impurities interferences were minimized by collecting a KBr background spectrum for each analysis. Spectra were recorded in the 500–4,000 cm^{-1} range. Absorption in the IR region takes places due to the rotational and vibrational movements of the molecular groups and chemical bond of a molecule. The two fundamental vibrations are stretching, where the atoms stay in the same bound axis but the distance between atoms increases or decreases, and deformation, where the positions of the atoms change relative to the original bound axis. The identification of absorption bands was based on the reported values available.

3. Results and discussion

3.1. Storm water composition and treatment performance

Small changes in chemical composition were noticed in the storm water runoff during four months of storage (Table 1). The filter bed porosity per L of filter beds is shown in Table 2. The average amount of organic matter measured as TOC was 600 mg L^{-1}, meanwhile the COD average was 1,400 mg L^{-1}. The pH was slightly acidic and the colour was high 8,300 mg Pt L^{-1}.

The pH of the wastewater passing through the columns filled with peat only remained in the same range of the inlet (4.0–4.5). As expected, the pH of the wastewater that passed through the column with peat and ash increased from 4.5 to 6.0. This might be due to the adsorption of tannic acids and humic acids to the filter material as well as the release of calcium oxide from the ashes. Only slight increase in conductivity was observed in the effluent from peat/ash column indicating minor release of metals and ions from ashes.

Columns filled with peat and ash mixture showed better performance than peat alone for wood leachate treatment. The TOC removal was found to be about 23 g kg^{-1} filter materials (peat and fly ash) as shown in Fig. 2. The increase of column height from 40 to 80 cm, however, contributed only slightly to the overall removal efficiency (TOC removal increased from 18 to 23 g kg^{-1} when column height increased from 40 to 80 cm). Columns filled with only peat showed less sorption potential as well as slower sorption kinetics.

Both filter beds showed good sorption potential for TOC removal. It should be noted that the breakthrough was reached almost at the same liquid to solid (L/S) ratio (~100) for all columns. However, the filter bed with mixed peat and ash was saturated after removal of 18–23 g TOC per kg filter, meanwhile the peat filter bed was saturated after removal of only 11–18 g TOC per litre filter bed. This result indicates that peat and ash mixture has higher potential in removing organic molecules than peat alone. This difference in behaviour might have several possible explanations such as the change in pH mentioned above, as well as the amount of cations present in the ash. Cations are well known to interact with different humic substances (HSs) and enhance the sorption capacity due to the Fuoss effect [18]. Furthermore, peat and ash used in this study contain high amounts of calcium (8,410 mg/kg) besides other cations. Cations are known to interact with natural organics in two manners; site specific strong binding and weak binding which is the presence of counter-ions in the vicinity of the organic molecule or electrostatic attraction [19]. Weak binding is present in all molecules and increases with carboxylic acid content and the structural arrangement of these groups, whereas strong binding is very organic specific and is not always present. A broad variability with the source of HSs in interaction

Table 1
Characterization of inflow leachate (mean ± standard deviation) (n = 10)

COD (mg L^{-1})	1,400 ± 120	pH	4.4 ± 0.37
TOC (mg L^{-1})	600 ± 72	EC	228 ± 23
PP (mg L^{-1})	200 ± 15	Colour (mg Pt L^{-1})	8,300 ± 800

Table 2
Filter bed porosity per L of filter beds

Filter	(A) Water (mL) trickling between 0 and 300 min	(B) Water (mL) content trapped in the filter bed	(A + B) (mL)	Porosity (%)
Peat I	250	645	895	89
Peat II	281	697	937	94
Peat III	227	722	949	95
Average (SD)	252 (27.1)	688 (39.3)	940 (28.4)	93 (0.03)
Peat/ash I	228	549	776	78
Peat/ash II	175	571	746	75
Peat/ash III	132	501	632	63
Average (SD)	178 (30.4)	540 (49.5)	718 (80.6)	72 (0.08)

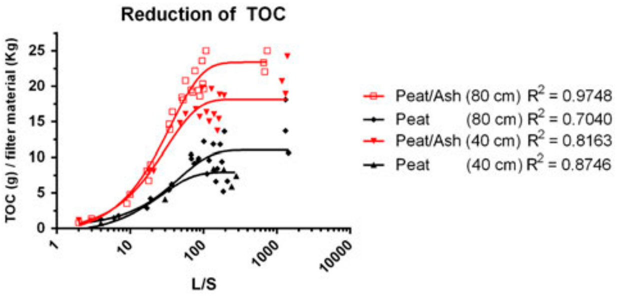

Fig. 2. Accumulated TOC reduction (g kg^{-1} filter) vs. L/S ratio. The filter height is shown within brackets (n = 3).

characteristics with calcium has been reported in literature [20]. Liao and Randtke [21] suggested that the complexation of natural organics with calcium might lead to stable complexes or aggregation. In another study, Chandrakanth and Amy [22] suggested that calcium preferentially interacts with oxygen-containing functional groups. The interactions with calcium vary the organic size depending on the organic content. The interaction of natural organics with cations result in various species, but due to the unknown equilibrium constants and the different interactions by various organics involving several mechanisms, the available data are limited.

The peat and ash mixture showed faster sorption/filtration of PP also, compared to peat alone (Fig. 3). The fact that peat is not efficient in removing DOC has also been reported by other researchers [23]. However, differently from the observed TOC removal, the saturation point for PP removal in the column with the mixture of peat and ash was achieved at a lower L/S ratio (~100) compared to the columns with peat alone (~600). Additionally, the total PP removed by both columns in terms of mass per mass (g/kg) was actually in the same range (around 10 g PP per kg of peat). Therefore, at the end of the process, basically similar sorption efficiency was achieved for both filters: peat alone and mixture of peat and ash. Moreover, since the same volume of peat + ash mixture has fewer pores than peat alone (Table 2), the hydraulic retention time (HRT) is expected to be higher than in the peat alone, which would promote higher contact time [24].

In different flow rates, both filter materials (peat and ash and peat alone) were saturated with the same amount of PP at the saturation point of the curve [25] (Fig. 3). This suggests that it is mainly the peat that adsorbs PP in higher amounts, while ash probably also adsorbs some other organic compounds existing in the wood leachate, as shown in Fig. 2. Since phenol-degrading bacteria have been observed in columns [12], pilot plant studies [15] as well as operating systems [26], it is also likely that these bacteria are present in both filters (peat + ash mix and peat). However, discrimination among pollutants reduced by sorption mechanisms, by mineralization due to bacteria metabolism and even by filtration not only has been the focus of this investigation. Low R^2 values due to high data dispersion were found particularly for TOC removal by peat columns (Fig. 2) in both lengths of 40 cm (R^2 = 0.6083) and 80 cm (R^2 = 0.7040), suggesting low performance of peat alone as filter material.

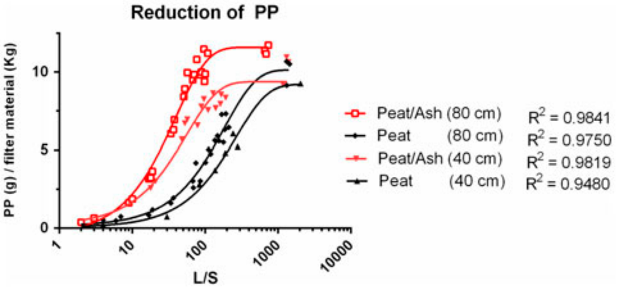

Fig. 3. Accumulated reduction of poly-phenols (PP) (in g per kg filter material) and liquid to solid (L/S) ratio. Column filter height showed within brackets.

The colour of the wastewater was highly correlated to the amounts of PP (R^2 = 0.91, Fig. 4). Therefore, colour might be used as a simple and cheap indicator of the treatment performance for PP removal in a full-scale filter. Also, COD and TOC are well correlated (Fig. 3) with a slope on 2.054 and an R^2 = 0.86, which makes it possible to use one parameter to calculate the other with reasonable accuracy.

3.2. FTIR analysis

FTIR was used to identify the surface groups of the sorbents and to identify those responsible for

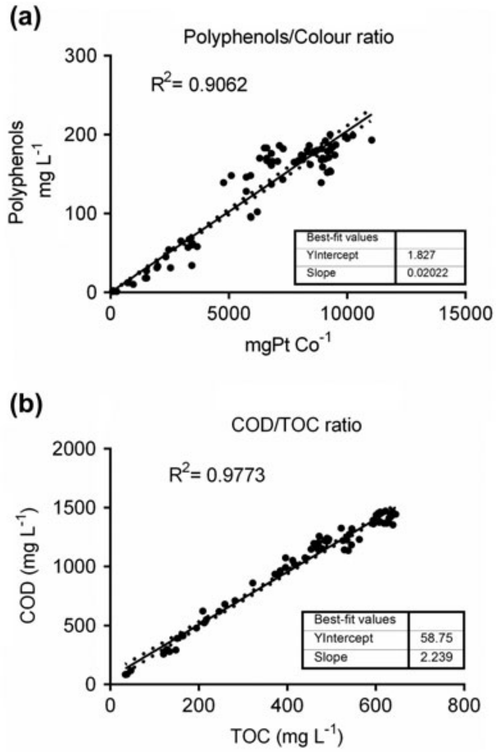

Fig. 4. Correlation between: (A) Polyphenols and Colour; (B) COD and TOC.

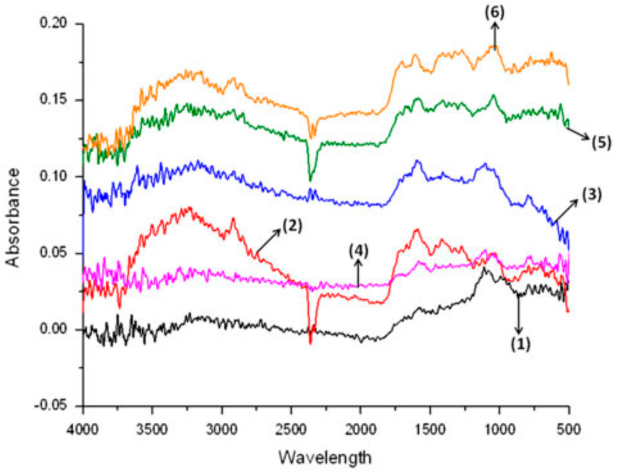

Fig. 5. FTIR spectra: (1) peat and ash; (2) column 2 (peat and storm water); (3) column 6 (peat + ash and storm water); (4) column 4 (peat + ash and storm water); (5) column 1 (peat and storm water); (6) peat.

Table 3
Peak assignments of absorption bands in FTIR spectra indicating functional groups

Wave no. (cm^{-1})	Assignment	Reference
3,621	Stretching of vibration of silanol SiO–H	[27]
3,448	H-bonded OH groups (alcohols, phenols, organic acids)	[28]
	H-bonded N–H groups	[29]
2,914	Methylene asymmetric stretch –CH_2	[30]
2,946	Methyl C–H asymmetric stretch –CH_3	[30]
2,861	Methylene symmetric stretch –CH_2	[30]
1,650	Aromatic and olefinic C=C, C=O of bonded conjugated ketones, quinone, C=O stretch of amide I	[30]
1,375	Symmetrical stretching of the carboxylate anion O–H deformation, C=O stretching of phenols, antisymmetric COO– stretching and aliphatic C–H deformations, salts of carboxylic acid and/or aliphatic CH N–O stretching nitrate	[28,31]
1,155	C–O stretching vibrations of C–O–C groups	[30]
1,043	Aromatic ethers, and possibly carbohydrates	[28]
	Si–O of silicates	[32]
898	Carbonate ion CO_3^{2-}	[30]
782	C–H deformation of substituted aromatic groups	[30]

binding pollutants found in the storm water. Comparisons between different FTIR spectra indicated the presence of the same basic FTIR bands in different samples with shifting or disappearance of some bands (Fig. 5). In the FTIR spectra of peat, various peaks were observed, such as 3,621, 3,448, 2,914, 2,946, 2,861, 1,700, 1,650, 1,600, 1,375, 1,155, 1,043, 898 and 782 cm^{-1} indicating the presence of various functional groups. The assignment of number to each wave has been explained in Table 3 [27–32]. In the case of peat and ash sample, some peaks were recorded in the range 700–1,200 cm^{-1}. The samples (peat only, peat and ash mixture) after coming in contact with storm water showed the shifting or disappearance of some peaks, which might be due to the change in the structure of functional groups, transformation or destruction of these groups due to the reaction with PP and other pollutants present in storm water. For example, most of the peaks disappeared in column 4 (peat and ash and storm water), while peak at 1,050 cm^{-1} disappeared in column 6 (peat and ash and storm water). On the other hand, column 1 (peat and storm water) showed a prominent peak at 1,050 cm^{-1} (attributed to the C=O stretch of polysaccharides) and column 2 (peat and storm water) exhibited a prominent peak at 2,915 cm^{-1} (due to asymmetric and symmetric stretches of aliphatic chains).

Different absorption bands have been identified in the FTIR spectra of different types of HAs which can be assigned to aromatic hydrocarbons, enolic aldehyde/ketone, carboxylate anions, carboxylic acids, intermolecular hydrogen bond (polymeric form) and aryl carboxylic acids, etc. The main absorption peaks which have been suggested by different researchers include H bonded OH, C=C of aromatic ring, C=O of carbonyl group (both carboxyl and ketonic), CH_2 and CH_3 groups [33,34]. These different functional groups play an important role during the sorption process. The possible sorption mechanisms which might involve include π–π interaction, where chemical bonding takes place between the –OH group of phenol and the carboxylic groups of the adsorbent's surface, so-called donor–acceptor complex, van der Waals forces and hydrogen bonding.

3.3. HRT in the filter bed

Peat is a highly porous material with many voids that are dependent on the degradation level of the peat [35]. The degradation level varies with the hydraulic conductivity [36]. In up-flow columns with continuous flow, the HRT is easily calculated by dividing the total void with flow rate. In the case of trickling filter, it is more difficult but it could be done by water flow or tracer technique as previously described [35]. However, when there is no constant inflow, these techniques do not apply. When dosing the fluid twice a day during one hour each time, different HRT can be found, since HRT changes gradually getting lower as the filter material becomes more and more saturated. It is also difficult to anticipate the interaction between the water that is flowing through the column and the water trapped in microspores or capillary spaces. According to Garzón-Zúñiga et al. [35], the water trapped in microspores is almost immobile, whilst the water trapped by capillary force interacts when flow occurs in the column. If one considers that around half of the water trapped in the column after trickling (Table 2) is bound to the filter by capillary force then the HRT estimated in the column could be around 12 h.

In earlier studies with similar filter material [16], the mixture peat and ash was also capable to remove metals to a high degree and chlorinated phenols to some degree. Metals are frequently considered as problematic in storm water runoff from wood storage areas [37]. Furthermore, many wood-based industries have used different chlorinated phenols as wood preservatives in the past. Nowadays, these compounds have often been found in soils of these industries [38]. Therefore, it is possible that these compounds could be found in the storm water.

4. Conclusions

The TOC removal efficiency of a filter composed by a mixture of peat and ash was approximately three times higher than the one achieved by a filter consisted of peat only in treating storm water contaminated with oak wood leachate. The mixture of peat and ash performed faster than peat only in the reduction of PP measured as tannins. However, regarding PP, both filters (peat + ash mixture and peat alone) achieved the same final removal at the saturation (breakthrough) point. FTIR analysis provided some relevant information about the functional groups (in peat and ash), which might possibly be involved in the sorption of pollutants in the storm water. However, due to the complex composition of both sorbents and storm water used in this investigation, more advanced analytical tools are required for better understanding of the mechanisms involved in the process.

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