

Organic Matter Removal from Leachate Using a Mixed Soil-Activated Carbon

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Abstract

A sustainable proposal was developed for environmental protection to the area around a landfill without control. Diverse columns for simulated permeable barrier packed with semi-wasted activated carbon (AC) from water treatment coming from a deionized equipment and soil samples taken from a landfill without any contamination control located in Mexicaltzingo (México) were studied to remove natural organic matter (NOM) from leachate samples (LS), taken from a landfill site, and laboratory prepared samples (LLS). Natural organic matter measured like chemical oxygen demand (COD), biochemical oxygen demand (BOD), nitrites and nitrates was removed through packed columns. Two different packed forms were tested; in column A, the AC was placed as a support for the landfill soil samples and, in column B, a homogeneous mixed of AC and landfill soil was used. Experiments showed that a short time, 60 min is optimal for a fast NOM removal, COD and BOD decreased up to 94% from the initial concentration in LS; for column B, the same parameters plus nitrites and nitrates were removed up to 100%, making it the most efficient treatment. There are not significant differences in NOM removal for soil samples taken at 3 or 12 meters and the used AC is a semi-wasted revalorized residue. The proposal in this work, for NOM removal through packed columns measured by 4 parameters, shows to be viable as an initial proposal to be used as a permeable barrier to reduce a contamination risk associated to the landfill area.

Keywords: Activated carbon; Leachate; Natural organic matter; Soil; Sustainable

Introduction

Commonly, organic matter (OM) is used to describe a mixture of heterogeneous chemical fractions [1]. So normally the term “natural organic matter (NOM)” is used for all the organic matter in a reservoir or natural ecosystem. NOM is formed by a wide variety of organic compounds that are primarily derived from the decomposition of plant and animal residues that contribute to offensive taste and odors [2,3]. Particularly, NOM from landfill leachates includes volatile fatty acids and refractory humic and fulvic-like compounds, depending on the stage of degradation in the landfill [3]. To measure NOM in synthetic conditions, some carbohydrates, hydrous metal oxides are used as standards to obtain an analytical signal to let follow NOM concentration in lab samples [2]. The main risk generated by NOM in soil and water is totally or partially diminished oxygen, thus the aquatic life and aerobic process are limited for nutrients [4]. As result, NOM is one of the major pollutant materials in leached liquids.

In Mexico, every year only 39% of 30 tons of municipal solid waste (MSW) was disposed of in a landfill [5]. In San Mateo Mexicaltzingo, which is a representative town located in central Mexico due to its commercial activity, there is an MSW disposal site that is outside Mexican Standards and environmental regulations [6-8]. Originally, sand mine has been used as a final disposal site for the last 20 years and is still in operation. The site covers 8705 m², of which approximately 5600 m² are in use. Among the material disposed at the site were pork meat residues and some other animal-derivates, resulting in high NOM content composed mainly humic-like materials, due to the age of the landfill [3].

Many studies focus on the removal of NOM with by different methods, such as ion exchange, membrane filtration, coagulation and adsorption in several materials [3]. Usually, activated carbon is used as a universal adsorbent for water and treated wastewater with heavy

metals, phenols, colorant and organic matter [1,9]. One disadvantage of organic matter removal with activated carbon is the large molecular size of the NOM, but a great advantage is that it is low cost, available and environment friendly [4]. In this work, we propose the use of semi-wasted activated carbon (AC) from deionized water equipment to reduce NOM from natural and synthetic leachate samples. The AC in deionized water equipment is replaced every 6 months and after it has no another use, therefore it was important for this work to revalorize this residue by testing a new use after his in end of life in these devices.

The stratigraphic profile shows that the soil in Mexicaltzingo is sandy [10] so NOM could seep through the soil into the phreatic zone, where drinkable water is extracted, becoming a risk for groundwater [4,11]. Unfortunately, in Mexico there are many sites without any type of contamination control where no real action is taken to diminish the generated pollution [8].

Permeable reactive barriers (PRB) have been used successfully to treat a wide range of contaminants [12]. A PRB consists of making a long trench around the soil and filling it with a reactive material, they are no toxic for humans and are functional to adsorb dangerous chemical compounds present in groundwater, and the system can be

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used with any type of soil. The principal limitation for PRBs is their slowness, this because they depend on the natural groundwater flow and are difficult to construct [13,14].

Laboratory assays using columns are an interesting option to establish the best operating conditions for pollutants control through the use of PRBs [15], where the results from these assays will allow the design of PRB for the landfill site in Mexicaltzingo town.

In recent years, studies have documented the huge technological problems involved in the restoration of aquifers polluted by organic compounds and this has led to an increase interest in new developments for controlling and preventing pollution by a wide range of contaminants [15-17]. This is specially the case for zones exposed to sources of contamination by spills or illegal dumping, etc.

The aim of this work was to study the reduction of organic matter through lab columns using a mixture of packed activated carbon intercalated with a sandy loam soil from the Mexicaltzingo landfill [18-20]. Also, to propose a sustainable treatment by, first, using a semi-wasted activated carbon from a water treatment device and, second, testing in columns the decrease of NOM in a landfill in Mexicaltzingo town.

Materials and Methods

Soil and leachate sampling

A stratigraphic profile from Mexicaltzingo was determined in a previous study [10]; some data from this profile are shown in Table 1. The profile shows that up to a depth of 60 m the predominant soil is sandy, at 3 and 12 m, the clay content is higher than the rest of the sample. So, for this research two types of soil samples (60 g) were collected at 3 and 12 m deep. The soil samples collected were air dried. Based on the textural classes described by the United States Department of Agriculture (USDA), there are two kinds of soil in this landfill: the first located at a depth of 3 m, is loamy sand and the second at a depth of 12 m is sandy.

A leachate sample (LS) was taken from a piezometric well of 60 m deep. The sample was collected in a polyethylene bottle and was used for the experimental removal test within 24 h after making the collection and the samples taken for COD were preserved with concentrated H₂SO₄. The volume collected for each sample was 2 L and they were taken during rainy season because the mobility of pollutants in water is higher than in dry season.

Leachate samples

COD, BOD, nitrites and nitrates solution:

1 L of leachate lab sample (LLS) was prepared and mixed with several standard solutions; 500 ml of 1000 mg/L for COD, 100 ml of 198 mg/L of BOD, 150 ml of 250 mg/L for nitrites (N-NO₂) and 250 ml of 100 mg/L for nitrates (N-NO₃). Each solution was prepared as follows:

1. COD solution: 0.851 g of C₈H₅KO₄ (Mallinckrodt 100%, standard primary) was dissolved in 1L of H₂O.
2. BOD solution: for the dilution water it was necessary to prepare a buffer with 8.5 g of KH₂PO₄ (Mallinckrodt, 99.2%),

21.75 g of K₂HPO₄ (Mallinckrodt, 99.8%), 33.4 g of Na₂HPO₄ (Baker JT, 98.1%), 1.7 g of NH₄Cl (Baker JT, 99.6%), all of which were dissolved in 1 L of distilled H₂O.

3. 1 L of the following solutions was prepared separately: 27.5 of CaCl₂ (Monterrey, 96%), 0.25 g of FeCl₃•6H₂O (Fermont, 97.9%) and 22.5 g of MgSO₄•7H₂O (Baker JT, 101.1%). 1 ml of each solution was taken to prepare 1 L of dilution water.
4. One capsule of polyseed NX (Interlab) was dissolved in 500 ml of H₂O (inoculum solution), 0.0750 g of C₆H₁₂O₆ (Fluka, 99.5%) and 0.0750 g of C₅H₉NO₄ (Fluka 99.5%) was dissolved in 500 ml of H₂O (GGA solution).
5. For BOD standard 20 ml of glucose-glutamic acid (GGA) solution and 16.7 ml of inoculum solution was mixed and filled to 1 L using dilution water.
6. Nitrites solution: 1.2320 g of NaNO₂ (Fermont, 99.6%) was dissolved in 1 L of distilled H₂O.
7. Nitrates solution: 0.7218 g of KNO₃ (Fermont, 99.9%) was dissolved in 1 L of distilled H₂O.

Column preparation and leaching test: For the experimental column only AC was used. This carbon comes from a water treatment system to produce water type 1 (conductivity <0.06 μS/cm) [21]. The activated carbon was recovered from the equipment, dried at room temperature, grounded and sieved with a 50 mesh (0.297 mm particles of AC). Glass columns were 1.8 cm × 30 cm (internal diameter x length) size.

First column was prepared packing 50 g of natural soil (3 or 12 m depth) taken from the landfill and at the top of the columns was placed 10 g of activated carbon (column A). A second column was prepared using a homogeneous mixture of 50 g of natural soil (12 m depth) and 10 g of activated carbon (column B).

Both column were oversaturated with water and allowed to drain the excess of water freely for 24 hours so humidity conditions were equivalent to field capacity [15]. Under this condition, the natural flow of the columns was 0.2 ml/min.

For leaching experiments, the lab and natural leachate samples were used separately. A volume of 100 ml of each sample (LS or LLS) was added to each column (A or B). After 1.5 h, a fraction of the leaching solution was collected (18 ml) continuously and taken to organic matter analysis (nitrites, nitrates, COD and BOD). The total time of column operation was 8 hours.

COD determination: The procedure for COD determination is described by NMX-AA-030-SCFI-2001 [22]. A hermetic tube is prepared by adding 1.5 ml of digestion solution A [K₂Cr₂O₇ (Mallinckrodt, 99%) solution 4.17 × 10⁻³ mol/L y HgSO₄ (Mallinckrodt)], 2.0 ml of leachate sample (LS or LLS) and 3.5 ml of silver acid solution [7.50 g of Ag₂SO₄ (J.T. Baker, 99.4%) in 500 ml of H₂SO₄ (Meyer, 98%)]. After properly mixed it, the chemical digestion can start by placing the hermetic tube in a HACH digester set up at 150°C during 2 hours. After the digestion and cooling down, the K₂Cr₂O₇ remained contained in the tube is titrated with SAF solution 0.025 eq/L [Fe (NH₄)₂(SO₄) • 6H₂O, Baker,

Soil depth (m)	Texture	pH	E.C. (dS/m)	C.E.C. (cmol/Kg)	Clay (%)	Silt (%)	Sand (%)
3	Sandy	5.9	1.42	7.52	5.94	12.62	81.44
12	Sandy clay	7.4	1.79	7.57	14.31	0.07	85.62

E.C.: Electric conductivity; C.E.C.: Cation exchange capacity.

Table 1: Characteristics of natural soil [10].

100.75%) previously standardized using a $K_2Cr_2O_7$ standard (Aldrich, 99.99%) potassium dichromate 0.025 eq/L.

BOD determination: The procedure for BOD determination is described by NMX-AA-028-SCFI-2001 [22]. A winkler bottle is prepared with an aliquot of leachate sample (LS or LLS) and filled with oxygen saturated water, previously prepared with an specific solution which contains 8.5 g of KH_2PO_4 , 21.75 g de K_2HPO_4 , 33.4 g de $NaHPO_4 \cdot 7H_2O$, 22.5 $MgSO_4 \cdot 7H_2O$, 0.25 g $FeCl_3 \cdot 6H_2O$, 27.5 g of $CaCl_2$ in 1.0 L of solvent and aired. The initial dissolved oxygen is measured by an oxygen dissolved calibrated meter YSI 5100 and then the winkler bottle is closed and incubated at 20°C for 5 days in a Memmert IPS749 incubator. At the end of the 5th day, the final oxygen dissolved can be measured in the same way.

Nitrites and nitrates determination: Nitrites were determined by NMX-079-SCFI-1987 [22] as follows; first a standard curve is prepared using $NaNO_2$ from 0 to 0.25 mg/L (0 to 3.6×10^{-4} mol/L). Then 10 ml of leachate sample (LS or LLS) without turbidity is placed in different tubes, to each tube 1.0 ml of sulfanilamine was added and left stand during 8 minutes, after 1.0 ml of NEDA solution [$C_{16}H_7NHCH_2CH_2NH_2 \cdot 2HCl$, Mallinckrodt, 98%] was added. After 60 minutes, absorbance was read at 543 nm in Thermo Evolution array equipment [21]. Nitrates were determined by ion electrode selective Thermo Scientific Orion, using a calibration curve prepared with KNO_3 solutions from 0.5 to 5 mg/L, and then following the common procedure of using an electrode method.

Results and Discussion

A leaching sample of the piezometric well was taken in a rainy season. The well was made to determine the stratigraphic profile as describe in section 2.1; the results for COD, BOD, nitrites and nitrates are shown in Table 2. The obtained values for the determined parameters correspond to middle-age landfill, between 10 and 20 years old [23] as a reference results are compared with Mexican standard limits. It is observed that the amounts of COD and BOD are high; therefore the leachate potentially contributes to the increase of NOM within the environment. These results are due to the municipal solid wastes (MSW) deposited in the landfill which degrade with time resulting in an important source of organic matter pollution in the site.

The characterization study of MSW for quartering method [24] is shown in Table 3. This characterization was made to identify a kind waste deposited in landfill where LS is produced. The sample tested and separated in this work was taken from 1 day of collected MSW is deposited in landfill; the main residue found was guts and feathers (47.2%). Overall, organic waste corresponds to 75% of the domestic

	COD	BOD	Nitrites	Nitrates
Concentration (mg/L)	1718	188	2.7	49.1
MS (mg/L) (2007)	20	10	1	10

Mexican standards (MS): [7].

Table 2: Leaching sample taken from piezometric well in Mexicaltzingo.

Subproduct	Weight (kg)	Composition percent
Guts and feathers	98.2	47.2
Food waste	57.4	27.59
Plastic	10.6	4.85
Paper and cardboard	9.2	4.422
Diapers	8.6	4.13
Glass	6.6	3.17
Others	17.4	8.6

Table 3: Quantity and composition of the Municipal solid waste generated in Mexicaltzingo town.

waste stream. Discarded plastic, paper, diapers, glass and other represented 25% of the MSW and other wastes, like textiles, wood, aluminum, etc., were also found but in less proportion. The high content of organic matter found in the MSW, which later is disposed in Mexicaltzingo's landfill, degrades and produces leachate for 20 years according with landfill age identify by leachate characterization, therefore, high quantities of these residues will increase the volume of leachate resulting in a higher probability of aquifers, land and plants polluted for a long exposition to generated NOM in site [25]. According with this result, MSW contributes to NOM in this site for 20 years, and NOM detected quantity is according with landfill age as the same other typical organoleptic properties like color, taste and odor also identified [17].

Figure 1 shows the results obtained for COD for simulated packed columns, it presents COD concentrations for soil samples taken at 12 meters deep for a) laboratory leachate sample (LLS) and c) leachate sample (LS) from the landfill and for the soil samples taken at 3 meters deep for b) LLS and d) LS. In all cases, it can appreciated the same behavior for all soil samples, a fast removal of COD during the first 60 min followed by a semi-stable quantity of COD until reach 350 min. For the leachate lab sample, COD decreased from 3485 mg/L to 115 mg/L during the first 60 minutes in the soil at 3 m deep, representing a decrease of 96% of the initial COD concentration. While for the leachate sample extracted at 12 m deep, it presented a decrease from 1718 mg/L to 912 mg/L, having only a 53% removal of the original COD concentration.

A characteristic of the tested leaching sample (LS) is that COD concentration corresponds to half of the laboratory prepared leachate (LLS). Samples originated from the piezometric well had also other chemical compounds like suspended and total solids, carbonates, fatty acids and refractory compounds as humic acids, which presence is consistent to the landfill's age and can be removed in the same column. While the prepared lab leachate sample only presented carbohydrates, hydrous metals, and others that simulated the NOM. These explain the higher COD removal in LLS than in LS; LLS samples had higher organic content and did not present a competition between all the others chemical compounds, like existed carbonates in LS [26].

Also, in the packed columns it can be observed an additional effect associated with the pH value; when the lab leachate sample passed through the packed column the pH value changed from 4.77 to 6.1,

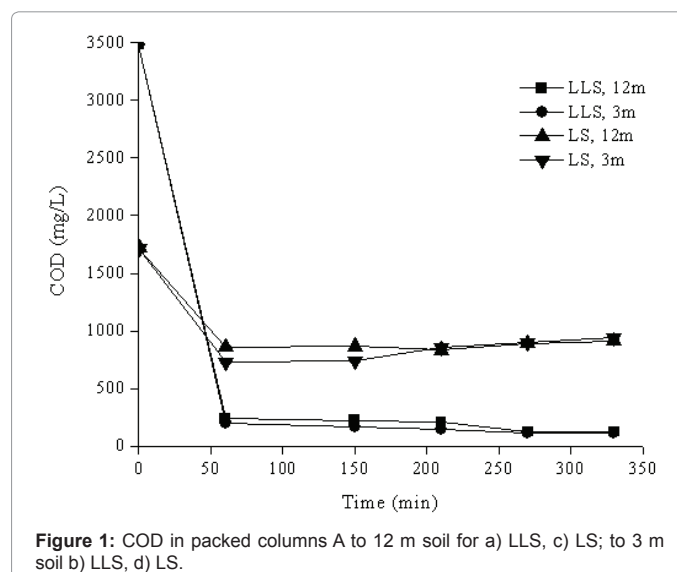


Figure 1: COD in packed columns A to 12 m soil for a) LLS, c) LS; to 3 m soil b) LLS, d) LS.

while the pH value for LS changed from 7.02 to 8.02 units. In both cases, a pH increase is due to organic matter decomposition and it is associated to microbial action since the packed columns were exposed to the light.

BOD results obtained for the soil sample taken at 12 meters deep for a) laboratory leachate sample (LLS) and c) leachate sample (LS) from the landfill and taken at 3 meters deep for b) LLS and d) LS, are presented in Figure 2. For the LS, after 60 min it is observed the initial BOD concentration decreased 50% at 12 m deep, while at 3 m deep it was possible to remove 80% of the initial BOD concentration. This shows that there is not real effect for BOD removal associated with the depth of the soil, deeper soil does not guaranty an increase in NOM removal. In the lab leachate samples, it can be observed the same behavior, which confirms that there is no effect in the soil depth; the removal percentage is higher in 3 m soil (94%) than in 12 m soil (60%). For longer times, up to 350 min, a different behavior is observed for both LS and LLS; removal of BOD in 12 m soil decreases at this time while the rest of the samples reach equilibrium at 270 min.

The carboxylic acids and phenolic compounds of the NOM had high molecular masses, high degree of aromaticity and, sometimes, formed complexes with other substances present in the water or adsorbent material. This factor is common when activated carbon is used to reduce NOM in water samples, and in this case, it explains the decreased adsorption of BOD in the packed columns with soil-activated carbon [17]. However, NOM removal in each column showed that the soil taken from Mexicaltzingo landfill acts like a natural permeable barrier against the organic matter pollution, expressed like COD and BOD. This effect is improved by adding activated carbon as support, which represents only 20% of the additional weight of the soil used for the packed columns. In the tested packed columns for COD and BOD removal, the saturation point was not reached with the sandy soil (3 m) or with the sandy clay soil (12 m). This last type of soil had a low exchange capacity that not had influence in the removal process, which confirms that there is not a real influence in NOM removal when using different soil samples of depths.

Table 4 shows the results obtained for nitrites and nitrates in the packed columns for LS and LLS. This results show that nitrites and nitrates were best removed in LS in all the columns. For example, nitrites in column A changed their initial concentration from 53.4

mg/L to 30.8 mg/L for LLS; removing a 43% of the initial concentration, while for LS the concentration changed from 2.7 mg/L to 0.98 mg/L; which is a 64% of nitrite removal. A similar behavior is observed for nitrates in both columns. For the LS, the column B removed 68% of the initial concentration (IC) which is a decrease from 49.1 to 15.3 mg/L, while in column A, concentration was decreased up to 15.3 mg/L which represents a 75% removal.

Nitrites and nitrates removal is influenced by the amount of NOM present [27], therefore the simultaneous removal of BOD and COD decrease the nitrites and nitrates elimination. The initial concentration of both does not affect the removal capacity of the packed columns.

Finally, the last packed column was prepared mixing soil extracted at 12 m and activated carbon (column B), results of the 4 measured parameters are shown in Figure 3 for the LS. The removal percentage for COD, BOD, nitrites and nitrates in LS is better in column B than in column A. For COD, the removal percentage reaches 93% of de initial concentration; this represents almost 1150 mg/L removed in column B, while column A removed 850 mg/L. For column B, the contact time increased since the flow that passes through the packed column was slower than in column A. For BOD, the removal percentage was 85%, a similar amount to the one obtained for column A, while for nitrates, the removal percentage reached was 99%.

Figure 3 shows the results for the packed column with mixed soil-AC. It is observed that NOM removal was much more effective than in the packed column with AC as upper support. NOM removal in the packed column with mixed soil-AC exceeds 90%, although the adsorption time is longer. This increased time is due to the high molecular weight of NOM, which slows the flow though, the mixed column where the presence of different size particles exists. However, this increase in time

Column	Nitrites				Nitrates			
	LLS		LS		LLS		LS	
	IC=53.4 mg/L		IC=2.7 mg/L		IC62.4 mg/L		IC=49.1 mg/L	
	Removal		Removal		Removal		Removal	
	mg/L	percent	mg/L	percent	mg/L	percent	mg/L	percent
A	30.8	43.4	0.98	64	36.8	41.1	17	65.4
B	12.1	77.4	0.9	67	15.3	75.5	15.3	68.9

Table 4: Nitrites and nitrates in laboratory and leaching sample removed in four types of columns for 60 min.

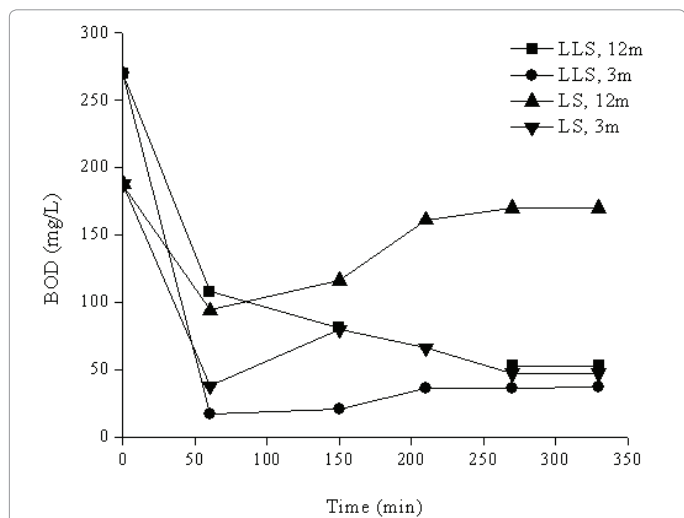


Figure 2: BOD concentration in packed columns A for 12 m and 3m soil for LLS and LS.

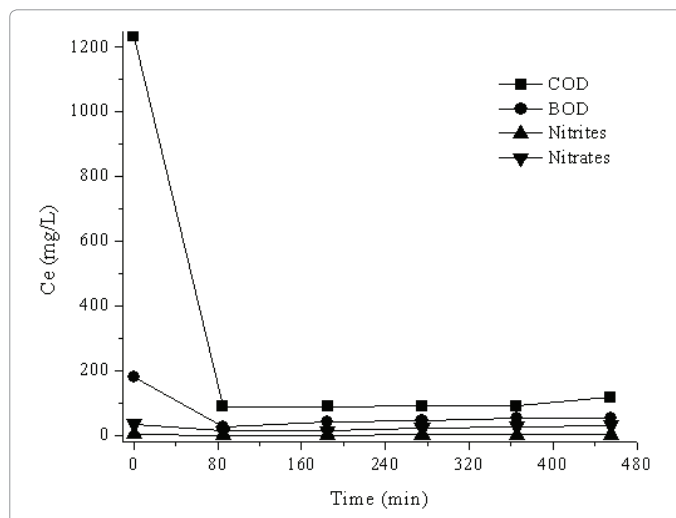


Figure 3: COD, BOD, nitrites and nitrates concentrations in soil plus activated carbon packed column for leachate sample (LS).

allows a higher contact time of the NOM with the soil surface and AC resulting in higher removal. Comparing the removal efficiency of NOM in columns A and B, removal rate is higher in column B for almost all parameters; very close to 100%. This high NOM removal in column B indicates that NOM is probably adsorbed in the surface of the soil and the activated carbon. Also that during this process there were not any electrostatic interactions between the samples and the activated carbon, which can exist in the AC support in column A [28]. Another possible mechanism is hydrogen bonding, which results from the combination on hydrophilic functional groups in NOM, like carboxylic groups present in the NOM [4]. The packed columns A and B show the same behavior for the 4 measured parameters at short intervals of time. It is observed a quick removal and equilibrium is reached at 480 min. The removal of contaminants, expressed as NOM, is due to the presence of activated carbon in each column.

Considering that the NOM content removed by the extracted soil depends on its structural properties (size, bonds, etc.) of the chemical compounds and the proposed experimental conditions, it is necessary to take into account the type of material to use in the column (whether as support or mixed with soil) and the appropriate amounts of these materials in order to project an effective permeable reactive soil barrier for pollution control.

Conclusions

Packed columns with sandy soil, sandy-clay soil and used activated carbon for NOM removal expressed as COD, BOD, nitrites, nitrates show that, at short periods of time (60 min), it is possible to achieve a removal higher to 60%, for both natural and laboratory prepared leachate. NOM removal is not affected by the type of soil but it is influenced by the type of column packing; being the best a mixture between soil-AC. The tested columns show that a mixture of 80% soil and 20% AC is enough to create a permeable reactive barrier that controls organic pollution in an unrestricted landfill site. These results show that it is possible to valorize used activated carbon for NOM treatment in leachate generated at a landfill, and in a future implementation as a permeable barrier.

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