

Stabilization of Paved Roads Using Geosynthetics

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EXECUTIVE SUMMARY

Geosynthetics have been used as reinforcement inclusions to improve pavement performance. While there are clear field evidences of the benefit of using geosynthetic reinforcements, the specific conditions or mechanisms that govern the reinforcement of pavements are, at best, unclear and have remained largely unmeasured. Significant research has been recently conducted with the objectives of: (i) determining the relevant properties of geosynthetics that contribute to the enhanced performance of pavement systems, (ii) developing appropriate analytical, laboratory and field methods capable of quantifying the pavement performance, and (iii) enabling the prediction of pavement performance as a function of the properties of the various types of geosynthetics.

Geosynthetics have been used in pavement design to address the functions of separation, filtration, lateral drainage, sealing, and reinforcement. Specifically, geosynthetics have been used for separation in pavement projects to minimize intrusion of subgrade soil into the aggregate base or sub-base. Also, geosynthetics have been used to perform a filtration function by restricting the movement of soil particles from the subgrade while allowing water to move to the coarser adjacent base material. In-plane drainage function of a geosynthetic can provide lateral drainage within its plane. In addition, geosynthetics have been used to mitigate the propagation of cracks by sealing the asphalt layer when used in pavement overlays. Finally, geosynthetics have been used in flexible pavements for reinforcement, which is the main focus of this paper. While the reinforcement function has often been accomplished using geogrids, geotextiles have also been used as reinforcement inclusions in transportation applications. The geosynthetic reinforcement is often placed at the interface between the base and sub-base layers or the interface between the sub-base and subgrade layers or within the base course layer of the flexible pavement. This leads to lower stresses over the subgrade than in unreinforced flexible pavements.

The improved performance of the pavement due to geosynthetic reinforcement has been attributed to three mechanisms: (1) lateral restraint, (2) increased bearing capacity, and (3) tensioned membrane effect. The primary mechanism associated with the reinforcement function for flexible pavements is lateral restraint or confinement. The name of this mechanism may be misleading as lateral restraint develops through interfacial friction between the geosynthetic and the aggregate, thus the mechanism is one of a shear-resisting interface. When an aggregate layer is subjected to traffic loading, the aggregate tends to move laterally unless it is restrained by the subgrade or by geosynthetic reinforcement. Interaction between the base aggregate and the geosynthetic allows transfer of the shearing load from the base layer to a tensile load in the geosynthetic. The tensile stiffness of the geosynthetic limits the lateral strains in the base layer. Furthermore, a geosynthetic layer confines the base course layer thereby increasing its mean stress and leading to an increase in shear strength. Both frictional and interlocking characteristics at the interface between the soil and the geosynthetic contribute to this mechanism. Consequently, the geogrid apertures and base soil particles must be properly sized. A geotextile with good frictional capabilities can also provide tensile resistance to lateral aggregate movement.

The aforementioned mechanisms require different magnitudes of deformation in the pavement system to be mobilized. In the case of unpaved roads, significant rutting depths (in excess of 25 mm) may be tolerable. The increased bearing capacity and tensioned membrane support mechanisms have been considered for paved roads. However, the deformation needed to mobilize these mechanisms generally

exceeds the serviceability requirements of flexible pavements. Thus, for the case of flexible pavements, lateral restraint is considered to contribute the most for their improved performance.

The results of field, laboratory and numerical studies have demonstrated the benefits of using geosynthetics to improve the performance of pavements. However, selection criteria for geosynthetics to be used in reinforced pavements are not well established yet. The purpose of this paper was to summarize information generated so far to quantify the improvement of geosynthetics when used as reinforcement inclusions in flexible pavement projects.

A Pullout Stiffness Test (PST) was recently developed at the University of Texas, Austin in order to quantify the soil-geosynthetic interaction in reinforced pavements. The equipment involves a modified large-scale pullout test modified to capture the stiffness of the soil-geosynthetic interface under small displacements. Research conducted using the PST has shown that monotonic pullout tests aimed at characterizing the soil-geosynthetic interaction under low displacements are promising. Although these pullout tests did not replicate the cyclic nature of traffic load conditions, it simulated the interface transfer mechanisms between soil and geosynthetic reinforcements that are expected in the field.

An analytical model was proposed to predict the confined load-strain characteristics of soil-geosynthetic systems under small displacements using the results obtained from the PST. This approach takes into account both the confined stiffness (J_c) and ability of geosynthetic to mobilize shear or interlock (τ_y), which are two important parameters governing the performance of geosynthetic interfaces. The two parameters can be combined to define a unique coefficient of soil-geosynthetic composite (K_{SGC}) that characterizes the soil-reinforcement interface. This coefficient is computed as:

$$K_{SGC} = 4 \cdot \tau_y \cdot J_c \quad (1)$$

A comprehensive field monitoring program is under way to relate the field performance to laboratory PST results for a number of geosynthetic reinforcements. While ongoing field monitoring is still in progress, good agreement has been obtained so far between the field performance and the properties defined from PST testing. Thus, a new performance-based test method in the form of a pullout stiffness test is promising as a performance-based test to evaluate the soil-geosynthetic confinement.

An overall assessment of the various tests developed so far for geosynthetic-reinforced pavements indicates that unconfined tests are simple, economical and expeditious, although they do not capture the important aspects associated with confinement and the type of soil. Also, unconfined tests have provided only index measures of the actual mechanisms, requiring subsequent correlations with field performance. It should be noted that field studies sometimes led to performance trends that contradicted the trends obtained using properties from unconfined tests. Accordingly, and based on the current body of literature, unconfined tests are considered inadequate for assessment of the performance of geosynthetic-reinforced pavements.

Previous research has led to a reasonably good understanding of the benefits achieved with the use of geosynthetics in pavement design but, for the most part, only from the empirical point of view. That is, while methods have been developed for designing geosynthetic-reinforced flexible pavements, quantification of the reinforcement mechanisms, identification of properties governing the pavement performance and, ultimately, acceptable design guidelines are yet unavailable.

Efforts are currently under way in the US to develop design models consistent with the AASHTO and mechanistic-empirical (M-E) approaches. The TBR and BCR ratios have been used in the AASHTO approach but are limited because the approaches are specific to the products and test conditions under which these ratios have been calibrated. Thus, M-E methods are considered more generic and, consequently, more promising as framework to incorporate the use of geosynthetics in current pavement design. However, due to the complex nature of flexible pavements, research to identify and quantify the properties governing the performance of reinforced pavements and its incorporation into M-E design is still under way.



The available literature involving field and laboratory test results is conclusive in that the mechanical properties of the geosynthetics used for pavement applications are improved under the confinement provided by the soil. Field test sections showed improved performance in the reinforced sections over the unreinforced sections in terms of reduced surface deflections. Overall, available experimental evidence indicates that the improved performance of geosynthetic-reinforced pavements can be attributed to lateral restraint mechanisms. Attempts have been made to quantify the lateral restraint in terms of the interface shear stiffness property of the soil-geosynthetic system.

A number of confined laboratory tests have been recently developed with the objective of quantifying the interface shear stiffness of the soil-geosynthetic system. Several of these tests have applied cyclic loads to the soil-geosynthetic system in an attempt to simulate the dynamic nature of traffic-induced loading. However, probably due to the fact that measurements are sensitive to small changes in displacements, currently available methods have resulted in significant scatter in test results. This has compromised the repeatability of the approaches and has made it difficult to differentiate the performance among different geosynthetics. Ongoing research focusing on confined testing under low displacements using monotonic loading pullout stiffness test appears promising to quantify relevant mechanisms in pavement reinforcement design.

Overall, it may be concluded that significant advances have been made in the area of geosynthetic reinforcement of pavements. While the state of practice is rapidly improving, further research is still needed to provide a better theoretical basis to the currently available empirical design approaches.

Effect of Geomembrane Thickness on Diffusion of Toluene

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ABSTRACT

The effect of thickness (1.0 mm, 1.5 mm, 2.0 mm, and 2.4 mm) on the diffusion of toluene through four high density polyethylene (HDPE) geomembranes extruded from the same resin, antioxidant package, and carbon black is examined and the diffusion and permeation coefficients are reported. The implications are discussed.

1. INTRODUCTION

Geomembranes are a common barrier in municipal solid waste liner systems to prevent the leakage of landfill leachate. HDPE geomembranes are widely used in this application because they are excellent barriers to advective flow and the diffusive transport of inorganic contaminants present in the leachate (Rowe et al. 2004). However, geomembranes are susceptible to diffusive transport of volatile organic compounds, which are commonly found in landfill leachate as by-products of the waste degradation (Kim et al. 1995). These contaminants are able to move through the HDPE geomembrane on a molecular level (Park and Nibras 1993; Prasad et al. 1994; Sangam and Rowe 2001, 2005; Edil 2003), resulting in potential contamination of surrounding soil and water.

Molecular transport through a geomembrane occurs in three steps: (1) adsorption of the contaminant onto the geomembrane, (2) diffusion through the geomembrane, (3) desorption from the geomembrane into the receptor fluid (Park and Nibras 1993; Prasad et al. 1994; Sangam and Rowe 2001, 2005). These processes are impacted by a variety of factors related to the geomembrane, test conditions, and contaminant types (Rowe 1998). For example, the diffusive flux through a geomembrane can change depending on properties of the geomembrane (crystallinity, degree of cross-linking, density, thickness, etc), permeant properties (molecular weight, polarity, initial concentration, etc.), and temperature (Michael and Bixler 1961, Rowe 1998, Sangam and Rowe 2001, 2005).

Ewais and Rowe (2014) studied the effects of the blown film process on the properties of geomembranes, and found that for different pulling speeds, there are differences in the mechanical and physical properties associated with blown-film geomembranes with exactly the same resin, but different thicknesses. Slight differences in crystallinity were reported for these geomembranes. Several studies (Michaels and Parker 1959; Michaels and Bixler 1961; Islam and Rowe 2009) have shown that increasing crystallinity of the geomembrane results in a decreased diffusion coefficient as diffusion occurs through the amorphous zone of the polymer. The objective of this study is to investigate the effect of geomembrane thickness in the diffusion process. The rate of diffusion should be inversely proportional to the square of the geomembrane thickness if the diffusion coefficient is constant (Cussler 1997).

2. MATERIALS AND METHODS

2.1 HDPE Geomembrane

The geomembranes used in this study were produced from the same resin and formed by blown-film extrusion. However, each geomembrane was pulled at a different speed from the dye, resulting in four different thicknesses. The properties of these geomembranes are shown in Table 1.

Table 1: Selected properties^a of geomembranes tested

Property	Unit	1.0	1.5	2.0	2.4
Thickness	mm				
Density	g/cm ³	0.946	0.946	0.947	0.946
Crystallinity – 1 ^b	%	53.1 ± 1.2	50.5 ± 0.7	46.0 ± 2.0	41.0 ± 2.7
Crystallinity – 2 ^b	%	51.3 ± 4.7	48.5 ± 2.3	45.7 ± 2.5	41.7 ± 2.5
Crystallinity – 3 ^b	%	50.1 ± 1.8	41.6 ± 7.4	39.2 ± 4.0	39.8 ± 3.2
Crystallinity – 4 ^b	%	53.6 ± 1.8	48.0 ± 1.6	46.7 ± 0.9	48.4 ± 1.5

^aEwais and Rowe (2014)

^bASTM E793

2.2 Organic compounds

The contaminant used in this study was toluene (Table 2). Toluene is a chemical commonly found in landfill leachate, and has health risks associated with exposure above the recommended level (Rowe et al. 2004, USEPA 2009).

Table 2: Selected properties^a of toluene

Property	Value	
Molecular weight	92.14	g/mol
Density	0.8669	g/cm ³
Molar volume	106.28	cm ³
Aqueous solubility ^b	515	mg/L
Log K ^c _{ow}	2.79	-

^aMontgomery and Welcom (1990), Sangam and Rowe (2001)

^bAt 20°C

^c*n*-octanol:water coefficient

2.3 Analytical Methods

Sample concentrations were analysed using Solid-Phase Micro Extraction gas chromatography with flame ionization detection. Each analysis used reference standards of known concentration to develop a calibration curve and sample concentrations were measured by comparison with the area under the curve for the test specimen with that for the standard.

2.4 Test Procedures

Diffusion tests were conducted using stainless steel cells (McWatters and Rowe 2009) at 21°C. A geomembrane separated the source and receptor of the cells. Volumes and initial concentration used are shown in Table 3 for each cell. The compartments were filled with deionized water and 1 ml of 5000 ppm BTEX in methanol was added to the source compartment.

The cells were sampled daily for the first week of the test and sampling frequency gradually decreased to once a month by 100 days into the test. When samples were taken, the same volume of deionized water replaced the volume extracted to ensure no air space developed in the cells.

Table 3: Diffusion test cell volumes and initial concentrations

Geomembrane thickness (mm)	Source Volume (mL)	Receptor Volume (mL)	c_0^a (ppm)
1.0	210	110	23.8
1.5	250	110	20.0
2.0	250	110	20.0
2.4	260	95	19.2

^aInitial concentration

The basic theory for diffusion through a geomembrane is summarized below, based on Sangam and Rowe (2001). The diffusive flux, f [$ML^{-2}T^{-1}$], through a geomembrane can be calculated using Fick's first law.

$$[1] f = -D_g \frac{dc_g}{dz}$$

Where: D_g is the diffusion coefficient of the compound through the geomembrane ($ML^{-2}T^{-1}$), c_g is the concentration of the compound in the geomembrane [ML^{-3}], and z is the length of the path of diffusion or the thickness of the geomembrane [L]. At any point through the thickness of the geomembrane, the change in concentration with respect to time can be expressed using Fick's Second Law:

$$[2] \frac{\partial c_g}{\partial t} = D_g \frac{\partial^2 c_g}{\partial z^2}$$

At equilibrium, the concentration of a contaminant in the geomembrane, c_g , can be related to that in the adjacent aqueous solution, c_f , by a relationship analogous to Henry's Law, viz:

$$[3] c_g = S_{gf} c_f$$

where: S_{gf} is the partitioning coefficient [-], and c_f is the solution contaminant concentration [ML^{-3}]. The diffusive flux through the geomembrane can be calculated using the fluid concentrations by substituting equation 3 into equation 1.

$$[4] f = -D_g \frac{dc_g}{dz} = -S_{gf} D_g \frac{dc_f}{dz} = -P_g \frac{dc_f}{dz}$$

Where: the product of the partitioning coefficient and the diffusion coefficient is expressed as P_g , the permeation coefficient [L^2T].

Theoretical diffusion curves were calculated and fitted to the experimental results to infer the D_g and S_{gf} of the contaminants using the finite layer contaminant transport model POLLUTEV7 (Rowe and Booker 2004). These curves were calculated using the above diffusion equations with finite mass boundary conditions as described by Sangam and Rowe (2001).

3 RESULTS AND DISCUSSION

Figure 1 shows the calculated and experimental diffusion profile of toluene through the 1.0 mm and 1.5 mm geomembranes while Figure 2 shows the calculated and experimental profiles through the 2.0 mm and 2.4 mm geomembranes. The calculated profiles use S_{gf} and D_g (using POLLUTEV7) that best-fit the experimental data (Table 4).

The equilibrium concentrations of the contaminants in the 1.0 mm geomembrane tests were slightly higher than for the other thicknesses. The concentration of toluene at equilibrium was approximately 25% of the initial concentration for the 1.0 mm thickness diffusion test, which reached equilibrium in the shortest time. The equilibrium concentrations for the other three thicknesses were approximately 18% of the initial concentration. More time was required to reach equilibrium as the thickness increased, showing that increased thickness did provide a beneficial effect in reducing transient contaminant transport.

The diffusion coefficients varied with the change in thickness from 1 to 1.5 to 2.0 mm (there is no practical difference between the D_g results for 2 and 2.4 mm). When the calculated diffusion coefficients were related to the geomembrane crystallinities (Figure 3) there was a distinct downward trend. This was due to the less tortuous path and available with a larger amorphous zone corresponding to the lower crystallinity (Michaels and Parker 1959). The difference in crystallinity for each thickness was caused by different thermal and stress histories experienced by the geomembrane during the manufacturing process (Ewais and Rowe 2014).

The rate of transport through the geomembrane was controlled by Fick's second law (Eq.2). However, when comparing the transport of toluene through these geomembranes of different thickness both the thickness and diffusion coefficient were increasing (Table 4). Thus the expected benefit of slowing the rate of mass transfer for a thicker geomembrane in terms of a lower D_g/H^2 (Table 4) was somewhat smaller than would be expected for a constant D_g . For example, if the D_g was constant at the value for the 1mm geomembrane then for the 2.4 mm geomembrane D_g/H^2 would only be $3.3 \times 10^{-6} \text{ s}^{-1}$ rather than the $5 \times 10^{-6} \text{ s}^{-1}$ (i.e., 33% smaller).

The steady state flux through the geomembrane is given by Ficks first law (Eq. 4) which will be proportional to the permeation coefficient, P_g (Table 4), and inversely proportional to the geomembrane thickness for a given difference in concentration across the geomembrane. Thus, it will be proportional to the ratio of P_g/H . Because of the different stress and thermal histories of the geomembranes of different thicknesses tested, the ratio of P_g/H is similar for the 1 and 1.5

mm thick geomembranes. Increased thickness beyond 1.5 mm resulted in a distinct decrease with increasing thickness and the lowest flux would be for the 2.4 mm thick geomembrane. However, again the benefit of increased thickness is smaller than if the geomembranes had the same diffusion coefficient. For example, if the D_g was constant at the value for the 1mm geomembrane then for the 2.4 mm geomembrane D_g/H would only be 1.2×10^{-8} m/s rather than 2×10^{-8} m/s (i.e., 40% smaller).

Table 4: Partitioning, diffusion, and permeation coefficients calculated using POLLUTEv7

Thickness, H (mm)	S_{gf} (-)	D_g (m^2/s)	D_g/H^2 (s^{-1})	P_g (m^2/s)	P_g/H (m/s)
1	150	1.9×10^{-13}	19×10^{-6}	2.9×10^{-11}	2.9×10^{-8}
1.5	200	2.3×10^{-13}	10×10^{-6}	4.6×10^{-11}	3.1×10^{-8}
2	190	2.8×10^{-13}	7×10^{-6}	5.3×10^{-11}	2.7×10^{-8}
2.4	180	2.7×10^{-13}	5×10^{-6}	4.9×10^{-11}	2.0×10^{-8}

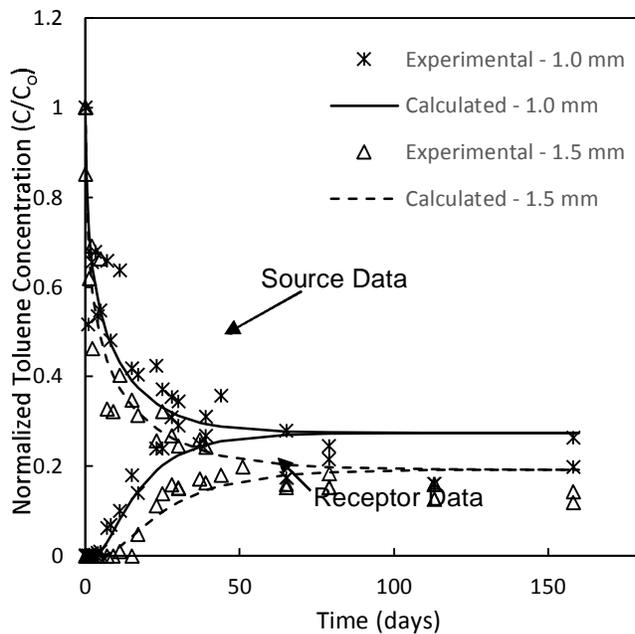


Figure 2 – Diffusion Plots for 1.0 mm and 1.5 mm thick geomembranes

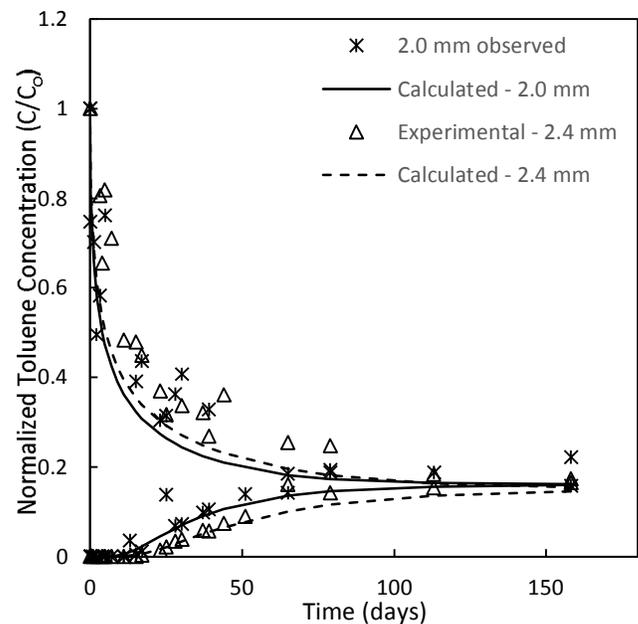


Figure 1 – Diffusion plots for 2.0 mm and 2.4 mm thick geomembranes

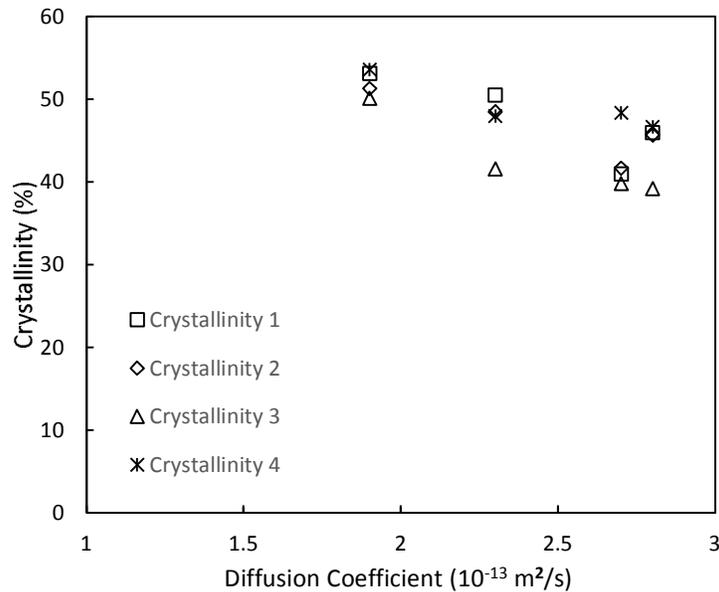


Figure 3 – Diffusion coefficient with respect to geomembrane crystallinity

4. CONCLUSION

This study examined the effects of the blown film process on migration of toluene through four different thicknesses of geomembranes produced with the same resin, but with different pulling speeds giving rise to the different thicknesses. Diffusion tests were conducted at room temperature (21°C) using stainless steel diffusion cells. S_{gf} and D_g were estimated using POLLUTEV7 to produce a theoretical diffusion curve that best fit the experimental data.

The diffusion coefficients of toluene through the four geomembranes showed a clear correlation with the crystallinities of the geomembrane; higher crystallinity corresponded to a lower diffusion coefficient. Although the four geomembranes had exactly the same resin, they did not have the same crystallinity because of the different stress and thermal histories (Ewais and Rowe 2014) and this resulted in change in the diffusion coefficient with changing thickness. Thus, the expected inverse relationship with thickness squared (H^2) does not hold because the thicker geomembrane has a lower crystallinity and hence a higher diffusion coefficient than the thinner geomembrane. However, while the full H^2 effect was not realized, there was still a slower rate of diffusive mass transport (and a longer time to equilibrium) with increasing geomembrane thickness.

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Diffusion of phenolic compounds through geomembranes

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ABSTRACT

This paper presents experimental results of the diffusion of phenolic compounds through a flexible polypropylene (f-PP) geomembrane, two co-extruded ethylene vinyl alcohol (EVOH) geomembranes, and a high density polyethylene (HDPE) geomembrane. The study considers the partition and diffusion of two chlorophenol contaminants (2,4,6-trichlorophenol and 2,3,5,6-tetrachlorophenol) that are known to be toxic even at very low concentrations. The concentration dynamics in the source and receptor chambers of the diffusion cells were interpreted using the numerical code Pollute. No significant differences could be noted between the various HDPE geomembranes, whether or not a thin EVOH film was co-extruded with HDPE. Diffusion through the f-PP geomembrane was significantly larger, by an order of magnitude, when one focuses on the permeation coefficient.

1. INTRODUCTION

Landfills contain micropollutants which can have toxic effects (acute toxicity, genotoxicity, reproductive toxicity etc.) (Sisinno et al., 2000; Takigami et al., 2002). Indeed, the presence of organic contaminants in the leachate from municipal solid waste landfills has been clearly demonstrated in several countries (Oman and Hynning, 1993; Ahel and Tepic, 2000; Robinson et al., 2001; Hiroshi et al., 2002).

The most frequently encountered organic micropollutants in leachate are monoaromatic hydrocarbons (benzene, toluene, ethylbenzene, xylene) and polyaromatic (naphthalene, phenanthrene etc.). Some chlorinated solvents (trichlorethylene, chloromethane etc.), plasticizers (phthalates, bisphenol A, etc.), pesticides and phenolic derivatives are also found in leachate (Harmsen, 1981; Bauer and Herrmann, 1997; Staples et al., 1998; Gron et al., 1999; Christensen et al., 2001; Cousins et al., 2002; Kjeldsen et al., 2002; Niederer and Goss, 2008, Van Praagh et al., 2011).

Phenolic compounds, especially certain derivatives of halogenated phenolic compounds, are known to be toxic to humans and the environment, even at very low concentrations. These compounds are used as disinfectants, biocides, preservatives, dyes, pesticides and organic chemicals in medicine and industry (Ramamoorthy and Ramamoorthy, 1997; Kujawski et al., 2004; Otero et al., 2005).

Geomembranes are widely used in geoenvironmental applications where they act as barrier to water and contaminants. A number of studies regarding the use of geomembranes focused on the diffusion of sodium chloride (Rowe et al., 1995) or volatile organic compounds (VOCs) for virgin HDPE geomembranes (Park and Nibras, 1993; Prasad et al., 1994; Müller et al., 1998; Sangam and Rowe, 2001; Park et al., 2012) virgin PVC, LLDPE with and

without a co-extruded ethylene vinyl-alcohol (EVOH) inner core geomembranes (McWaters and Rowe, 2008; 2010), fluorinated HDPE geomembranes (Sangam and Rowe, 2005) and aged HDPE geomembranes (Rowe et al., 2003; Islam and Rowe, 2008; 2009).

Diffusion of phenolic compounds has also been recently studied for an HDPE geomembrane and HDPE films (Touze-Foltz et al., 2012, Mendes et al., 2014). However, to the authors' knowledge no data are available for other types of geomembranes like flexible polypropylene geomembranes (f-PP) or HDPE co-extruded geomembranes with an inner EVOH core. It is thus the objective of this paper to present the results obtained while studying the diffusion of 2,4,6-trichlorophenol, and 2,3,5,6-tetrachlorophenol with such geomembranes.

2. EXPERIMENTAL PROCEDURE

2.1 Geomembranes

The study was conducted using a 1.5 mm thick f-PP geomembrane representative of the f-PP geomembranes available on the European market that could be used in landfills as an alternative to HDPE geomembranes. The rate of crystallinity of the f-PP geomembrane was measured to be 12.2%.

The diffusive transport of phenolic compounds was also examined in two co-extruded geomembranes with a 0.05-mm-thick layer of EVOH. Both geomembranes had HDPE outer layers and were 1.0 and 1.5 mm thick. Their rate of crystallinity was measured to be 50%. With its polar oxygen-hydrogen (OH) groups, EVOH has outstanding barrier properties against nonpolar gases such as oxygen, nitrogen, volatile compounds, and helium (Zhang et al., 1999). EVOH layers typically combines a highly ordered crystalline structure interspersed with disordered amorphous regions with high resistance against diffusion of gas and solvents (Zhang et al., 1999; McWatters and Rowe 2010). However, to the best of our knowledge, the diffusion of phenolic compounds in EVOH has not yet been studied.

The study was conducted using a 2 mm thick HDPE geomembrane representative of the HDPE geomembranes encountered in landfill bottom liners in Europe. Its crystallinity measured according to ASTM E 794 is 59.1%. This geomembrane is similar to the one used by Touze-Foltz et al. (2011) while studying the diffusion of VOCs.

2.2 Phenolic compounds

2.2.1 Phenolic compounds under study

Results of the diffusion of 2,4,6-trichlorophenol (2,4,6-TCP) and 2,3,5,6-tetrachlorophenol (2,3,5,6-TeCP) geomembrane are presented in this paper. Those compounds were included in a mixture containing 13 phenolic compounds: phenol, o-cresol (2-MP), p-cresol (4-MP), 2-chlorophenol (2-CP), 4-chlorophenol (4-CP), 2,4-xyleneol (2,4-DMP), 3,4-xyleneol (3,4-DMP), 2,4-dichlorophenol (2,4-DCP), 2,4,6-trichlorophenol (2,4,6-TCP), 2,3,5,6-tetrachlorophenol (2,3,5,6-TeCP), 2,3,4,6-tetrachlorophenol (2,3,4,6-TeCP), pentachlorophenol (PCP) and bisphenol A (BPA).

The selection of those phenolic compounds was performed taking into account the following criteria: (1) polarity, (2) solubility in water, (3) mobility of pollutants in soils, (4) presence in leachate, and (5) toxicity.

Concentrations were chosen based on two different elements: (1) a literature review to determine minimum, maximum and average values of concentrations of those phenolic compounds in leachate previously presented by Touze-Foltz et al. (2012) and Mendes et al. (2014) which is not repeated here; and (2) on the values of limit of detection (LOD) and limit of quantification (LOQ) calculated using the method developed by Limam et al. (2010) to analyze these compounds via the headspace solid-phase micro-extraction (HS-SPME) coupled with gas chromatography-mass spectrometry (GC-MS) which was used in this paper. Concentrations chosen for the methylphenols are slightly larger than average values found in leachate, since values encountered in the literature are significantly lower than the LOQ.

2.2.2 Preparation of the solutions

In a volumetric flask, 0.01 g of each compound was mixed with 0.1 g of phenol, o-cresol and p-cresol and 1 g of BPA in absolute methanol to obtain 100 ml of a stock solution. The desired amount of this stock solution was introduced in the bottles in the case of partitioning tests and in the source of the diffusion cells in the case of diffusion tests. The bottles and diffusion cells were subsequently filled with de-ionized distilled water (DDW) to obtain the desired concentration.

A preliminary test evidenced a biodegradation of some of the phenolic compounds under study during the batch partitioning and diffusion tests. It was thus decided to add a biocide agent (400 mg/L HgCl₂) to limit the effects of biodegradation.

2.3 Analytical methods

The quantification of phenolic compounds was performed by solid phase micro-extraction gas chromatography - mass spectrometry (SMPE-GC-MS) using the analytical procedure developed by Limam et al. (2010). The detailed protocol can be found for example in Mendes et al. (2014).

2,4,6-trichlorophenol-¹³C was used as internal standard for the quantification of 2,4,6-TCP, and pentachlorophenol-¹³C₆ was used as internal standard for the quantification of 2,3,5,6-TeCP.

The procedure took place in two steps: a derivatization and a headspace extraction.

In the derivatization step 5 ml of aqueous sample was introduced in a 20 ml PTFE-capped glass vial. Sodium chloride at 40% (W/V), 200 mg of KHCO₃ and 30 µl of acetic anhydride were added. A pre-incubation step of 5 min. at 80°C was then necessary to obtain a complete derivatization reaction prior to the extraction step.

Then the headspace extraction of target compounds was performed with a 100 µm PDMS SPME fiber at 80°C for 30 min with agitation (500 rpm). Afterwards, the SPME fiber was desorbed in the injector at 250°C for 5 min. GC/MS analysis was then performed as previously described.

Phenolic compounds quantification was performed using the single ion monitoring mode. The ions m/z 196 and 232 were used respectively for the 2,4,6-TCP and 2,3,5,6-TeCP.

3. TEST PROCEDURES

3.1 Control cell

Control tests were performed in a cell identical to those used for the batch partitioning test and the diffusion tests to assess the losses that may occur during the test due to chemical/cell material interaction as no geomembrane is introduced in those tests.

3.2 Batch partitioning test

Batch partitioning tests were performed at room temperature (i.e. $23 \pm 1^\circ\text{C}$) in 120 ml glass bottles equipped with screw-tight Teflon lined caps for sampling. The bottles were covered with an aluminum foil in order to avoid photo-oxidation. The experimental procedure followed was grounded onto the one described by Islam and Rowe (2009). In preparing the geomembranes for batch partitioning experiments, the HDPE geomembranes were cut into pieces (30mm x 20mm) weighing 6g in total. According to Nefso and Burns (2007) cutting the geomembrane into smaller pieces does not affect the ultimate sorption capacity, because organic/polymer equilibrium sorption is a dissolution controlled process, and not a surface controlled process.

The geomembrane pieces were immersed in solutions at the concentration used in the diffusion test. Initial concentrations of 2,4,6-TCP and 2,3,5,6-TeCP were equal to $10 \mu\text{g/L}$.

Two bottles were filled with osmosed water and a mixture of phenolic compounds. One of the two bottles without geomembranes was used as control test.

Concentrations of the various phenolic compounds were measured initially and after a period of 157 days which is, based on previous experiments, long enough to reach equilibrium in the bottles.

3.3 Calculation of partition coefficient

The concentration of contaminant in the geomembrane c_g and in solution c_f are linked according to Equation 1 (Henry's Law):

$$c_g = S_{gf} c_f \quad (1)$$

Where S_{gf} is the partition coefficient and is dependent on temperature, fluid, geomembrane, and contaminant of interest. The partitioning coefficients S_{gf} were calculated for each phenolic compound according to Equation 2 adapted from Sangam and Rowe (2001):

$$S_{gf} = \frac{((c_{f0} V_{f0})(1 - p_s) - c_{fF} V_{fF} - \sum c_i V_i) p_g}{M_g c_{fF}} \quad (2)$$

Where c_{f0} and c_{fF} were respectively the initial and final concentrations of the solution (g/L), V_{f0} and V_{fF} were respectively the initial and final volumes of the solution (L), p_s is the proportion of contaminant sorbed on glass determined based on the quantification of concentration evolution in B3 and B4 test, assumed to be independent of

the presence of a geomembrane specimen or not, ρ_g is the geomembrane density (g/L) and M_g is the initial mass of geomembrane (g).

3.4 Diffusion test

3.4.1 Experimental procedure

A permeation/diffusion method in which the geomembrane divides a testing cell into two compartments was used as this method simulates more closely the transport process that is expected in a landfill application than immersion/sorption tests (Sangam and Rowe 2001).

During the tests, chemical concentrations from both source and receptor were monitored with time. A single test was performed at $23^\circ\text{C} \pm 1^\circ\text{C}$ in a glass cell.

The diffusion cell consists of two 0.12m internal diameter glass cylinders terminated by rounded ends. A metallic bridle is used to assemble both parts of the cell that are identical. The geomembrane specimen is located between the two parts of the cell. No sealant is used. This system allows a very quick test setup. Tests can be started immediately after installation of the geomembrane into the cell. Each part of the cell contains a sampling port with a Teflon cap and a second port that can be used for the filling of the cell. Cells are lying in a horizontal position while the tests are performed. It is similar to the test cell previously used by Touze-Foltz et al. (2011, 2012). Those cells were especially designed to minimize the time of test set up. The volume of each chamber is equal to 0.73L. The cells were covered with aluminum foil in order to avoid photo-oxidation.

3.4.2 Calculation of diffusion coefficient

The diffusion of organic compounds through an HDPE geomembrane can be modelled by Fick's first law:

$$f = -D_g \frac{dc_g}{dz} \quad (3)$$

Where: f is the mass flux or permeation rate per unit area ($\text{g}/\text{m}^2/\text{s}$), D_g is the diffusion coefficient of organic compounds through the geomembrane (m^2/s), c_g is the concentration of compound in the geomembrane (g/L), and z is the distance parallel to the direction of diffusion (m). The change in contaminant concentration at any point in the geomembrane with time, t , is governed by the following differential equation according to Fick's second law:

$$\frac{\partial c_g}{\partial t} = D_g \frac{\partial^2 c_g}{\partial z^2} \quad (4)$$

If the source and receptor fluids are similar, the flux associated with the diffusion process can be obtained by substituting Eq. (1) into Eq. (3):

$$f = -P_g \frac{dc_f}{dz} \quad (5)$$

Where P_g is the permeation coefficient or mass transfer coefficient (m^2/s).

The test approach was based on concepts and theory proposed by Rowe et al. (1995), Sangam and Rowe (2001), and Islam and Rowe (2009) for geomembranes. For these closed systems, the mass of contaminant in the source solution at any time t is equal to the initial mass minus the mass that diffused through the geomembrane and can be written as:

$$c_t(t) = c_0 - \frac{1}{H_s} \int_0^t f_t(\tau) d\tau \quad (6)$$

Where: $c_t(t)$ is the concentration of contaminants in the source solution at time t (g/L), c_0 is the initial concentration in the source solution (g/L), H_s is the height of source fluid (volume of source fluid per unit area) (m), $f_t(t)$ is the mass flux of contaminant into the geomembrane at time t ($g/m^2/s$). The concentration in the receptor compartment at any time, $c_b(t)$, can be expressed similarly as:

$$c_b(t) = c_{b0} - \frac{1}{H_b} \int_0^t f_b(\tau) d\tau \quad (7)$$

Where $c_{b0}(t)$ is the initial concentration in the receptor solution (g/L), H_b is the height of the receptor (volume of receptor chamber per unit area) (m), and $f_b(t)$ is the mass flux of contaminant into the receptor chamber at any time t ($g/m^2/s$).

The diffusion (D_g) and partition (S_{gf}) coefficients were deduced following the procedure described by Sangam and Rowe (2001) using the finite layer analysis program POLLUTE v7© (Rowe and Booker, 1997).

4. RESULTS AND DISCUSSION

4.1 Control cells

4.1.1 Blank sorption tests

No significant sorption was noticed on glass for the two phenolic compounds under study in the bottle which did not contain geomembrane pieces.

4.1.2 Blank diffusion tests

Figure 1 shows the variation in phenolic compounds concentration as measured during the test period in the control cell. Concentrations did not exhibit a significant decrease along time for the two phenolic compounds under study.

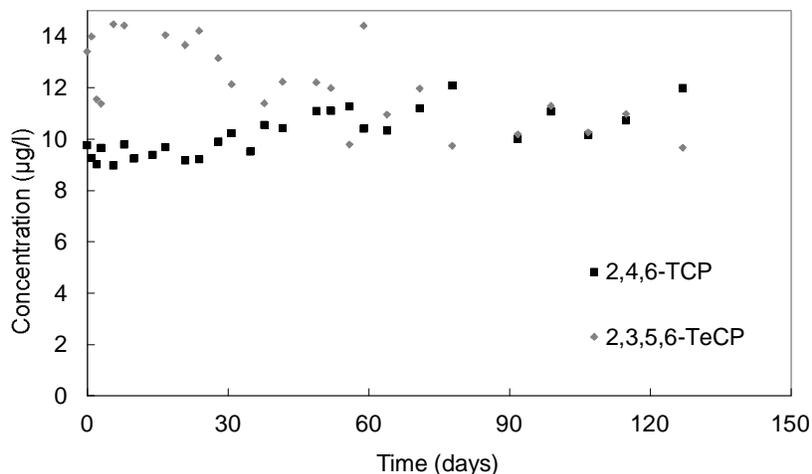


Figure 1. Observed concentrations of compounds in control cells for diffusion tests

4.2 Partition test

S_{gf} values for each phenolic compound were calculated assuming that the mass loss onto glass would occur even in the presence of a geomembrane specimen, following the methodology described in Section 3.2. The S_{gf} values of 2,4,6-TCP and 2,3,5,6-TCP are presented in Table 1. The value of the partition coefficient obtained for the HDPE geomembrane is one order of magnitude smaller than the value of the partition coefficient obtained for the other geomembranes. Similar figures are obtained for f-PP and co-extruded geomembrane when one focuses at 2,3,5,6-TeCP, while the partition coefficient of 2,4,6-TCP is larger in the f-PP geomembrane as compared to both co-extruded geomembranes.

4.3 Diffusion test

Figure 2 shows the variation in concentration with time of 2,4,6-TCP in the source chamber. No concentration of 2,4,6-TCP was detected in the receptor chamber along the 126 days of testing, probably in relation with the large values of partitioning coefficients. The adjustment was thus only performed on the concentration measured in the source chamber.

As far as 2,3,5,6 TeCP is concerned, concentrations in the source and receptor chambers are presented. Only in the case of the f-PP geomembrane, probably in relation to the large partition coefficient, no concentration of 2,3,5,6 TeCP could be detected in the receptor chamber. Diffusion coefficients obtained are presented in Table 1. The diffusion coefficients in the HDPE and the f-PP geomembrane are one order of magnitude larger than the diffusion coefficient in the co-extruded geomembrane.

On the overall, while multiplying the partition coefficient and the diffusion coefficients, permeation coefficients obtained for the HDPE and the two co-extruded geomembranes are of the same order of magnitude. The permeation

coefficient for the f-PP geomembrane is one order of magnitude larger, probably in relation to a larger affinity of 2,4,6-TCP and 2,3,5,6 TeCP for the f-PP geomembrane than for the HDPE geomembrane.

As a consequence, the result previously obtained as regards the diffusion of VOCs in coextruded geomembranes, that it was significantly lower than the diffusion through a HDPE geomembrane, is no longer true when one focuses on the diffusion of phenolic compounds. Results obtained for a given contaminant cannot and should not thus be extended to a different contaminant.

Table 1. Synthesis of diffusion results

Geomembrane	f-PP	HDPE	Co-extruded EVOH 1mm	Co-extruded EVOH 1.5mm
2,4,6-TCP				
D (m ² /s)	1.2×10 ⁻¹³	1.5×10 ⁻¹³	0.5×10 ⁻¹⁴	4×10 ⁻¹⁴
Sgf (-)	504	18	265	103
Pg (m ² /s)	6.05×10 ⁻¹¹	2.7×10 ⁻¹²	4.1×10 ⁻¹²	2.7×10 ⁻¹²
2,3,5,6 TeCP				
D (m ² /s)	2.5×10 ⁻¹³	2.3×10 ⁻¹³	2×10 ⁻¹⁴	3×10 ⁻¹⁴
Sgf (-)	357	38.5	378	377
Pg (m ² /s)	8.93×10 ⁻¹¹	8.85×10 ⁻¹²	11.3×10 ⁻¹²	8.9×10 ⁻¹²

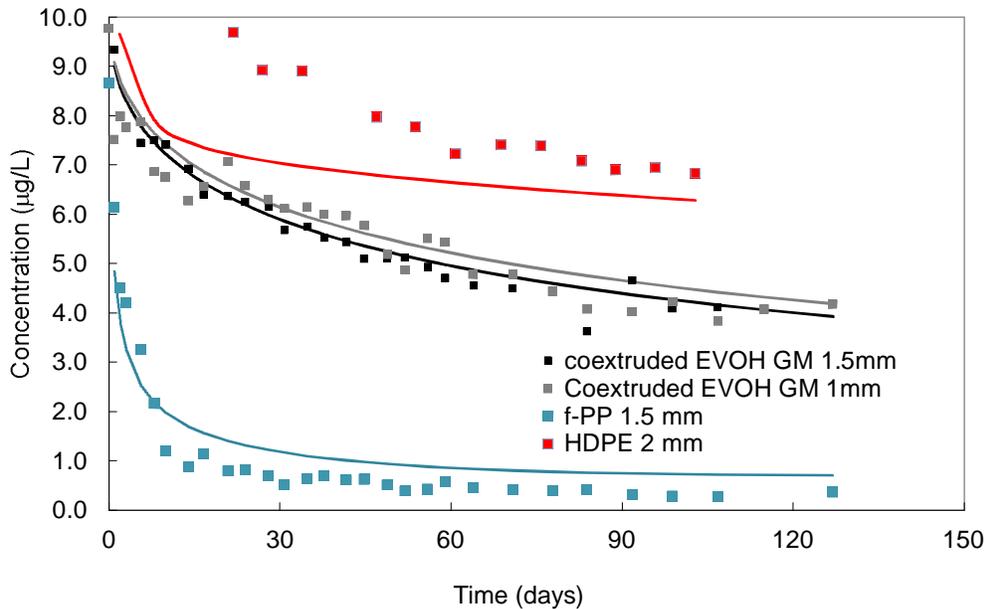


Figure 2. Observed concentrations and modelling for 2,4,6-TCP in the source chamber for the 4 geomembranes under study

5. CONCLUSIONS

The objective of this paper was to show the results of an experimental study performed in order to determine the values of diffusion coefficients of phenolic compounds in a f-PP geomembrane, a HDPE geomembrane and two co-extruded HDPE geomembranes with an EVOH inner core. Measurable concentrations were only obtained in the source chamber for 2,4,6-TCP and in both the source and receptor chamber for 2,3,5,6 TeCP, except for the f-PP geomembrane. The values of diffusion coefficients are very close in the f-PP and HDPE geomembrane, on the order of $10^{-13} \text{m}^2/\text{s}$, while they are one order of magnitude smaller for the two co-extruded geomembranes. This is balanced in the case of the HDPE geomembrane by a partition coefficient which is one order of magnitude smaller than the partition coefficient in the f-PP and the two co-extruded geomembranes. Finally, permeation coefficients in the HDPE and co-extruded geomembranes are very close to each other. The permeation coefficient for the f-PP geomembrane is one order of magnitude larger.

The conclusion thus given in the literature that co-extruded HDPE geomembranes with an inner EVOH core are more efficient towards the diffusion of VOCs than HDPE geomembranes cannot be extended to phenolic compounds.

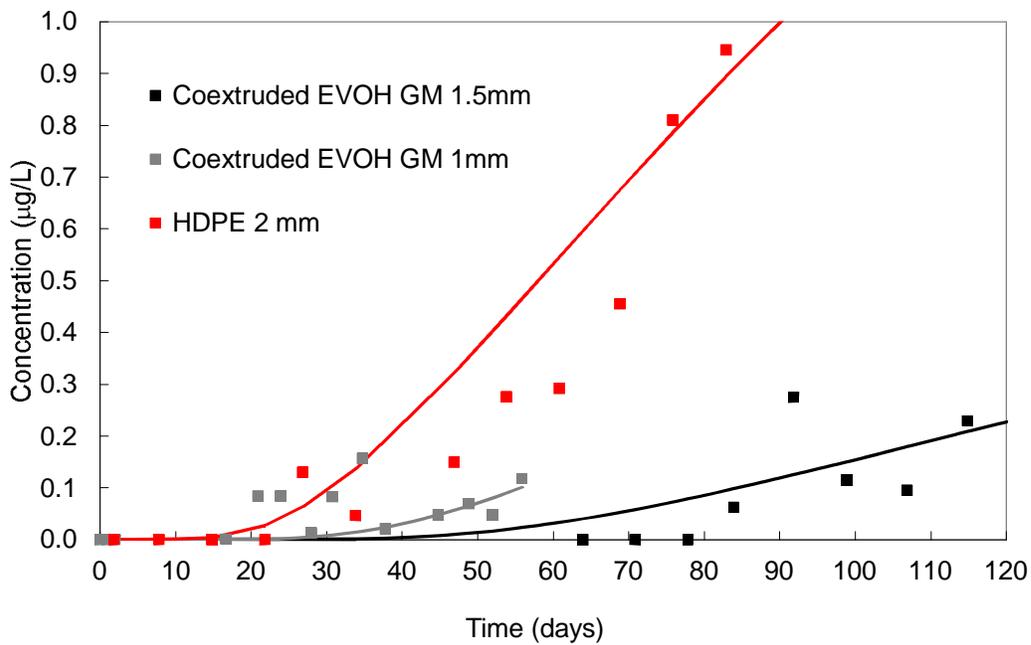
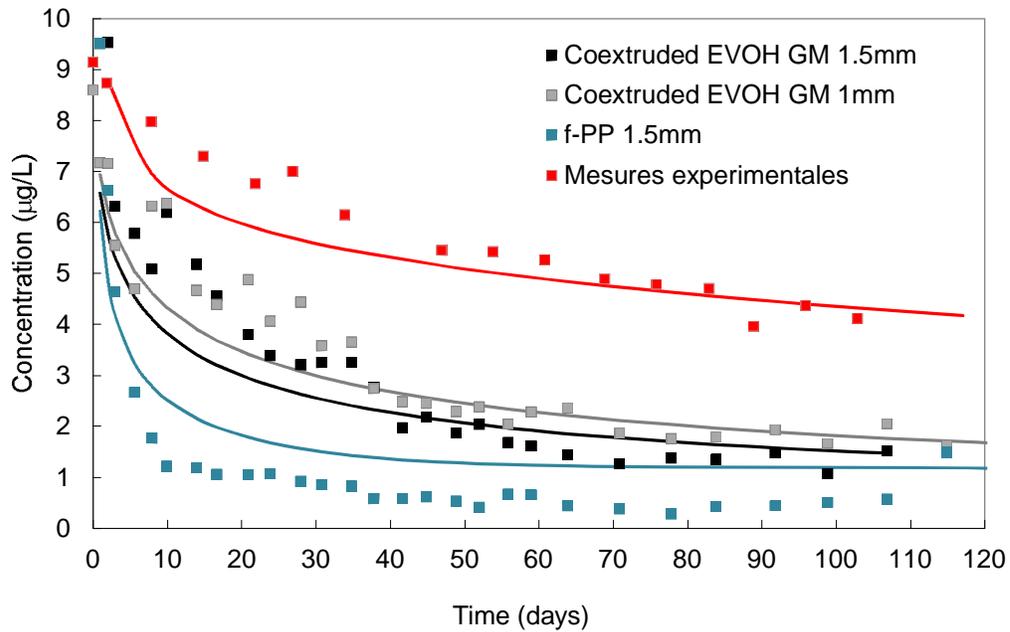


Figure 3. Concentrations in the source and receptor chambers for the diffusion of 2,3,5,6 TeCP

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