

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-xylenol (2,4-DMP), 3,4-xylenol (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 methyl-phenols 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):

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

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.

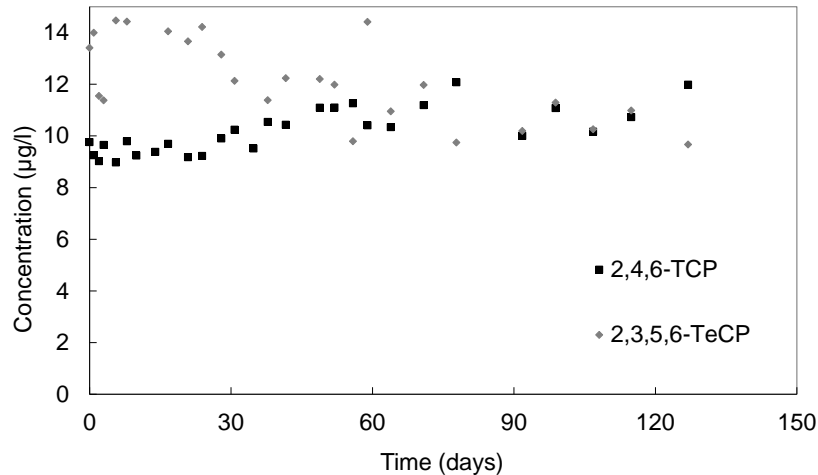


Figure 1. Observed concentrations of compounds in control cells for diffusion tests

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

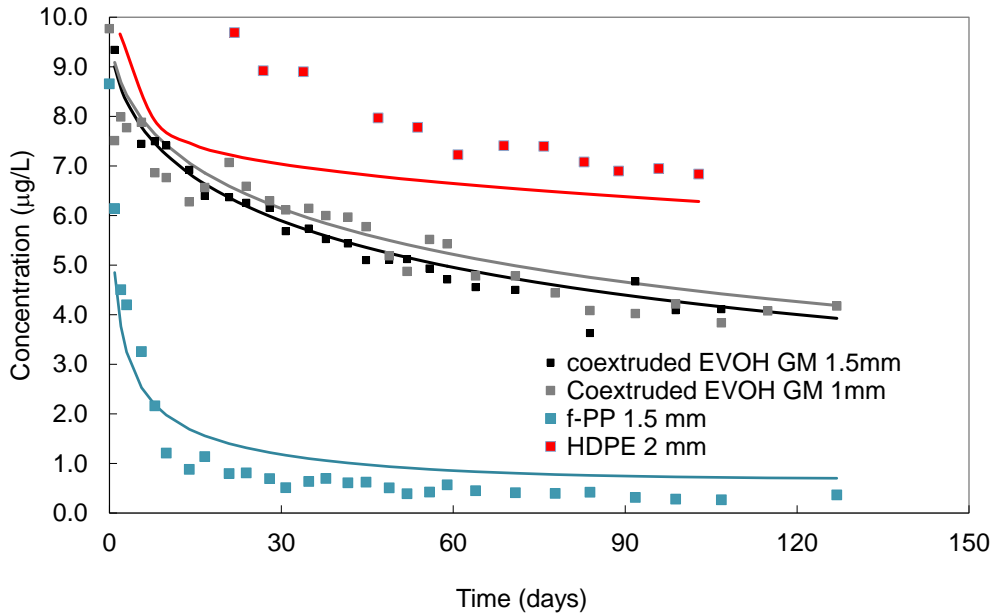


Figure 2. Observed concentrations and modelling for 2,4,6-TCP in the source chamber for the 4 geomembranes under study

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.

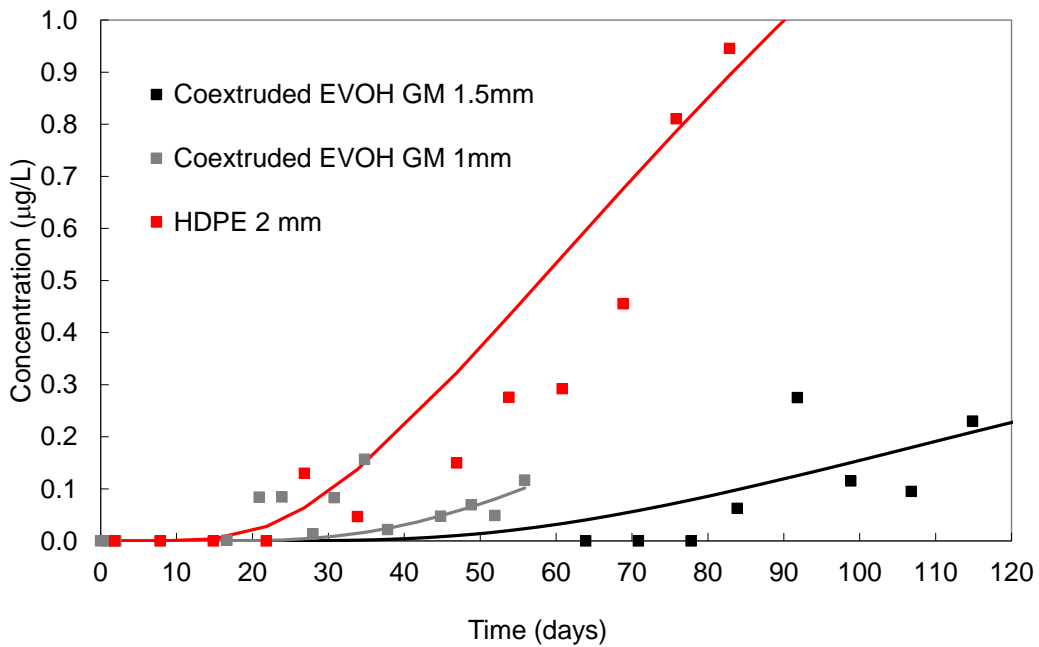
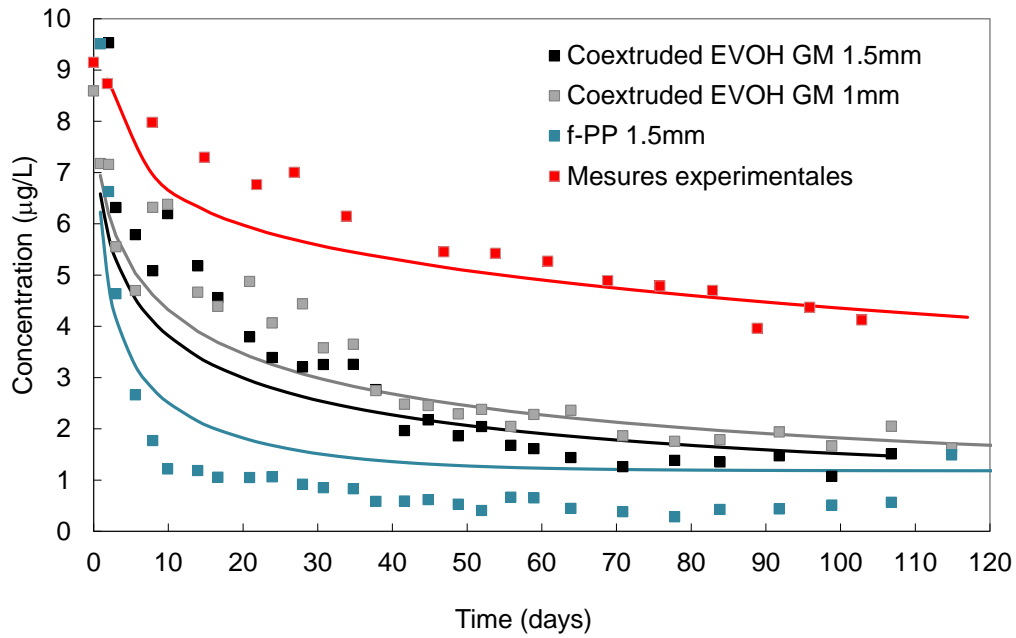


Figure 3. Concentrations in the source and receptor chambers for the diffusion of 2,3,5,6 TeCP

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