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# Sorption of chlorophenols on geotextiles of the geosynthetic clay liners and an HDPE geomembrane

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**Abstract :** Sorption tests were performed in the laboratory to quantify the sorption coefficient. This article describes the experimental results obtained regarding sorption on various constitutive geotextiles and bentonites of geosynthetic clay liners and high density polyethylene geomembrane of seven chlorophenols: o-chlorophenol, p-chlorophenol, 2,4-dichlorophenol, 2,4,6-trichlorophenol, 2,3,4,6-tetrachlorophenol and pentachlorophenol. The majority of halogenated phenol derivatives are known to be toxic even at very low concentrations. Sorption was studied through batch sorption tests once bentonite and geotextiles were separated from each other. The result obtained is that the sorption isotherms obtained are non-linear. As far as the partition coefficient are concerned, two different trends were observed, first for geotextiles which are nonwoven needle punched, and second for geotextiles which are woven. On the contrary no significant differences were observed between powdered bentonites; In this case of HDPE geomembrane, partitioning coefficients range from 2.65 to 205 respectively for 4-chlorophenol and pentachlorophenol. An increase in the partition coefficient and the permeation coefficient is observed with the increase in the number of chlorine atoms on the phenolic nucleus, probably related to the polarity of the molecules studied.

Key Words : Adsorption; Chlorophenols; HDPE geomembrane; Geosynthetic clay liners.

## 1. Introduction

Knowledge of organic micropollutant transfers in barrier seal materials from waste storage facilities is limited to volatile organic compounds [1-3]. This determination does not make it possible to evaluate the potential impact of other organic micropollutants such as phenolic compounds. This determination does not make it possible to evaluate the potential impact of other highly toxic organic micropollutants found in large quantities in the leachates of waste storage facilities, such as phenolic compounds from paints, solvents and cleaning products, plastics, petroleum product residues, pharmaceuticals, packaging of agricultural products, insecticides and treated wood in the waste. This is why it is interesting and necessary in the field of waste

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storage facilities to consider extending the study of the diffusion already carried out for certain volatile organic compounds to the phenol family. The choice of these phenolic micropollutants was made taking into consideration several criteria such as the mobility of pollutants in soils, their solubility in water, their polarity, their presence in leachates and their toxicity [4]. The objective of this study is therefore to evaluate the sorption 2,4-dichlorophenol potential of 2-chlorophenol, 4-chlorophenol, 2,4,6-trichlorophenol, 2.3.4.6tetrachlorophenol, 2,3,5,6-tetrachlorophenol, and pentachlorophenol on two different bentonites, two different geotextiles and one virgin HDPE geomembrane. Geomembranes are increasingly used in geo-environmental applications where they act as a barrier to water and contaminants. Several studies concerning the use of geomembranes have focused on the diffusion of sodium chloride [5] or volatile organic compounds (VOCs) on virgin HDPE geomembranes [6-9].

#### 2. Experimental Procedure

#### 2.1. Presentation of GCLs studied

Two geosynthetic clay coatings representative of the bottom coatings of municipal solid waste landfills were studied corresponding to GCL1 & GCL2 studied by Ahari et al. [4]. Their properties are summarized in Table 1.

GCL1 contains natural Wyoming sodium bentonite, while GCL2 contains two different calcium activated bentonites. The two GCLs consist of a woven geotextile, a nonwoven needled geotextile of both polypropylene (PP) and a layer of granular or powdery bentonite, all bonded and held by needling. GCL1 and GCL2, obtained from same manufacturer consist of GTX1 and GTX2.

Component	Property	GCL1	GCL2	
		PP nonwoven	PP nonwoven	
Cover geotestile	Nature	needle punched	needle punched	
Cover geolexine		(GTX1)	(GTX1)	
	Mass/unit area	$220 \text{ g/m}^2$	$220 \text{ g/m}^2$	
	Туре	natural sodium	activated calcium	
Pontonito		(BNT1)	(BNT2)	
Demonite	Form	powdered	powdered	
		$5.7 \text{ kg/m}^2$	6.1 kg/m <sup>2</sup>	
Carrier geotextile	Noturo	PP woven	PP woven	
	Inature	(GTX2)	(GTX2)	
	Mass/unit area	$110 \text{ g/m}^2$	$110 \text{ g/m}^2$	
GCL		needlepunched	needlepunched	

#### Table 1. Selected characteristics of GCLs

#### 2.2. HDPE geomembrane

The study was carried out using a 2 mm thick HDPE geomembrane with a crystallinity of 59.1%, representative of the HDPE geomembranes encountered in landfill bottom liners in Europe. The geomembrane used is the same as that used by Touze-Foltz et al. [11].

#### 2.3. Chlorphenols

Seven chlorophenols (CPs) were evaluated 2-chlorophenol (2-CP), 4-chlorophenol (4-CP), 2,4-dichlorophenol (2,4-DCP), 2,4,6-trichlorophenol (2,4,6-TCP), 2,3,5,6-tetrachlorophenol (2,3,5,6-TeCP), and pentachlorophenol (PCP).

Some common chemical properties of the selected cholrophenols are given in Table 2.

The octanol-water partition coefficient ( $K_{ow}$ ) of the pollutants under study in this paper strongly increases with the number of chlorine atoms whereas the water solubility (hydrophilicity) decreases. Furthermore, the degree of dissociation of chlorophenols increases (indicated by decreasing p $K_a$  values) with

increasing number of chlorine atoms. It is thus expected that the difference in properties of the various chlorophenols results in differences in sorption behaviour on the various components of GCLs tested in this study.

The selection of those chlorophenols was performed taking into account the following criteria: polarity, solubility in water, mobility of pollutants in soils, presence in leachate, and toxicity.

Concentrations of the contaminants were identical to the ones in the studies by Ahari et al. [4]. They were chosen based on two different elements: (a) a literature review to determine minimum, maximum and average values of concentrations of those chlorophenols in leachate presented in Table 3 [12-24]; and (b) on the values of limit of detection (LOD) and limit of quantification (LOQ).

Contaminants	Formula	Molecular weight (g/mol)	Solubility at $20^{\circ}$ C (g/l)	p <i>K</i> a	Log K <sub>ow</sub>
2-CP	C <sub>6</sub> H <sub>5</sub> ClO	128.56	27	9.37	2.53
4-CP	C <sub>6</sub> H <sub>5</sub> ClO	128.56	28	8.52	2.29
2,4-DCP	C <sub>6</sub> H <sub>4</sub> Cl <sub>2</sub> O	163.00	4.5	7.90	3.20
2,4,6-TCP	C <sub>6</sub> H <sub>3</sub> Cl <sub>3</sub> O	197.45	0.434	6.00	3.67
2,3,5,6-TeCP	C <sub>6</sub> H <sub>2</sub> Cl <sub>4</sub> O	231.89	0.100	5.02	5.02
PCP	C <sub>6</sub> Cl <sub>5</sub> OH	266.34	0.014	4.74	5.85

 Table 2. Selected properties of CPs tested

	Min. value	Max.	Mean	Corrected	LOD (ng/l)	<i>C</i> (µg/l)
Contaminants	(µg/l)	value (µg/l)	value (µg/l)	mean (µg/l)		
2-CP	0.003	0.510	0.107	0.06	16.47	10
4-CP	0.070	1.300	0.611	0.61	-	10
2,4-DCP	0.010	12.820	1.026	0.30	1.04	10
2,4,6-TCP	0.002	1.870	0.162	0.06	1.11	10
2,3,5,6-TeCP	0.012	0.012	0.012	0.01	1.15	10
PCP	0.015	21.610	3.798	0.83	1.07	10

#### 2.4. Analytical methods

The quantification of chlorophenols was performed using solid phase micro-extraction chromatography - mass spectrometry (SMPE-GC-MS) using the analytical procedure developed in our laboratory by Limam et al. [25]. The GC-MS was equipped with a Combi PAL autosampler allowing automatic SPME extraction. The GC split-splitless injector was operating in the Splitless mode.

The procedure took place in two steps: a derivatization and a headspace extraction. Clorophenolic compounds quantification was performed using the single ion monitoring mode. The ions m/z 162; 196; 202; 213; 232; 266; 274 were used respectively for the 2-CP; 2,4-DCP; 2,4,6-TCP; 2,3,4,6-TeCP and 2,3,5,6-TeCP and PCP. Then the headspace extraction of target compounds was performed with a 100  $\mu$ m 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.

#### 3. Test Procedures

#### 3.1 Batch adsorption test on geotextiles

The adsorption tests of chlorophenols on the geotextiles GTX-1 and GTX-2 were carried out in order to quantify the adsorption coefficients  $K_d$  of the phenolic compounds. The test consists of completely immersing 2 g of each type of geotextile cut into pieces in solutions with five different concentrations, in 100 ml glass bottles.

The solid / liquid ratio adopted for our experiments is 1/40, *ie* 2 g of geotextile for 80 ml of the solution containing the pollutants.

The solid / liquid ratio adopted in the experiments, is consistent with the ones adopted by Rowe [3] and Ganne [26] in works they performed regarding the sorption of VOCs on geotextiles.

The experiments were carried out in two stages, first of all determining the time of attaining equilibrium on two geotextiles and two concentrations.

First, a kinetic study was done to determine the time to reach equilibrium. Once the equilibrium time was determined, the adsorption tests of different geotextiles were carried out in different solutions at four different concentrations for the determination of isotherms (equilibrium sorption). Indeed all the tests are duplicated. Samples were taken at t = 0 to determine the initial concentration of phenols in the solution, followed by sampling at the end of the tests at t = 72h. All the experiments were carried out in the geosynthetic test laboratory whose temperature is close to  $23^{\circ}$ C.

#### 3.2. Batch adsorption test on bentonites

The adsorption tests of the chlorophenols on the different bentonites presented in Table 1 were carried out in order to quantify the adsorption coefficients  $K_d$ . The test consists in introducing 2 g of each sieve at 160 microns and dried at 60°C for 48 hours in various solutions (40 ml) at five different concentrations in 50 ml centrifuge tubes.

The solid / liquid ratio adopted in the experiments was 1/20. This ratio is consistent with the one adopted in the work performed by Banat [17] and Richards and Bouazza [27] regarding the sorption of phenol on bentonites.

In the same way as for geotextiles, two steps were carried out: a first step intended to determine the time to reach the sorption equilibrium and a second step to determine equilibrium concentrations

Preliminary kinetic experiments showed that adsorption equilibrium was reached within 48 h. At the end of the contact period the contents of the bottles were filtered, centrifuged for 3 h at 3000 tours.min<sup>-1</sup> using a Jouan C412 centrifuge and the supernatant was subsequently analyzed for residual concentration of chlorophenols.

#### 3.3. Batch Partitioning test for HDPE geomembrane

Batch partitioning tests were performed at room temperature (i.e.  $23 \pm 1^{\circ}$ C) in 120 ml glass bottles equipped with screw-tight Teflon lined caps for sampling. The bottles were covered with an aluminium foil in order to avoid photoxidation. The experimental procedure followed is grounded onto the one described by Islam and Rowe [28]. In preparing the geomembranes for batch partitioning experiments, the HDPE geomembrane were cut into four pieces. According to Nefso [29] 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.

Batch partitioning tests consist in immersing 6g of geomembrane, in solutions at the concentration used in the diffusion test  $10\mu g/l$  for chlorophenols. 4 bottles were used in total.

The bottles were filled with osmosed water and a mixture of phenolic compounds. B3 and B4 without geomembrane were used as control tests.

Concentrations of the various chlorophenols were measured each week in B1 and B2 in order to determine the time required to reach equilibrium. In B3 and B4 only one sampling was performed after 120 days of contact. According to the equilibrium time observed in B1 and B2, 80 days of equilibrium would have been sufficient.

The concentrations of contaminant in the geomembrane  $C_g$  and in solution  $C_f$  are linked according to Equation 1 (Henry's Law):

$$\mathbf{c}_{\mathrm{g}} = \mathbf{S}_{\mathrm{gf}} \mathbf{c}_{\mathrm{f}} \tag{1}$$

Where  $S_{gf}$  is the partitioning coefficient and is dependent on temperature, fluid, geomembrane, and contaminant of interest. The partitioning coefficients  $S_{gf}$  were calculated for each bottle and each phenolic compound according to Equation 2 adapted from Sangam and Rowe [9]:

$$S_{gf} = \frac{\left(\left(c_{f0}V_{f0}\right)\left(1 - p_{s}\right) - c_{fF}V_{fF} - \sum c_{i}V_{i}\right)\rho_{g}}{M_{g}c_{fF}}$$
(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,  $\rho_g$  is the geomembrane density (g/L) and Mg is the initial mass of geomembrane (g).

#### 4. Results and discussion

2,4-DCP

2,4,6-TCP

2,3,4,6-TeCP

2,3,5,6-TeCP

PCP

26.59

26.15

77.24

60.40

97.53

1.45

1.40

1.49

1.52

1.54

0.99

0.99

0.99

0.99

0.99

#### 4.1. Results of batch adsorption tests for geotextiles

The experimental results obtained for the determination of the adsorption parameters concerning the geotextiles are presented in the form of isotherms in Figure 1 for GTX1. Thus, the synthesis of the adsorption parameters of the different chlorophenols calculated for the various geotextiles is presented in Table 4

Sorption isotherms are non-linear contrarily to what was observed in the case of VOCs [3, 26]. The Freundlich model described by Eq. 3 was thus used to determine the sorption coefficient:

$$q_e = K_{\rm F} {\rm C_e}^{1/n}$$

Where:  $q_e$  and  $C_{eq}$  are the concentration in the solid in  $\mu g/g$  and the equilibrium solute concentration in  $\mu g/ml$ , respectively,  $K_F$  represents the sorption capacity, and 1/n is related to the energy distribution of the sorption sites.

The amount of each phenolic compound adsorbed at equilibrium,  $q_e$ , was calculated using Eq. 4:  $q_e = (C_0 - C_{eq}) \times V/W$  (4)

Where:  $C_0$  and  $C_{eq}$  (µg/l) are the liquid-phase concentrations of each organic compound at initial and equilibrium, respectively, V is the volume of the solution (l), and W is the masse of dry adsorbent used (g).

GTX1 GTX2 Freunlich linear linear Freunlich  $\mathbf{R}^2$  $\mathbf{R}^2$  $\mathbf{R}^2$  $K_{\rm d}$  $K_{\rm F}$ 1/n Kd  $K_{\rm F}$ 1/n R 2-CP 20.03 1.47 0.98 2.90 0.88 13.59 1.41 0.96 2.70 0.86 1.78 4-CP 1.55 0.98 90.32 0.97 30.59 3.00 0.87 2.80 0.83

0.90

0.92

0.90

0.90

0.90

7.62

12.13

21.93

47.57

61.79

1.24

1.28

1.28

1.48

1.47

0.97

0.97

0.98

0.99

0.99

3.00

3.50

6.00

5.90

7.60

0.86

0.89

0.95

0.91

0.92

4.00

4.50

8.90

6.40

8.40

Table	4. Synthesis of	partitioning coef	ficients for the geo	otextiles of the ch	nlorophenols und	er study at 23°C
		<b>I</b>				

396

(3)



Fig. 1. Results of batch adsorption tests for GTX1

The concentrations measured during these experiments were not corrected, since the blanks tests did not reveal any sorption of the phenolic compounds on the glass. The values of  $K_d$  partition coefficients of different phenols for different geotextiles are 2.7 to 8.9 ml/g and there is a slight difference between woven geotextiles and needled geotextiles. For comparison, these values remain low compared to volatile organic compounds (VOCs). As an example, the adsorption coefficient ( $K_d$  in ml/g) for benzene and toluene are: 32.2 and 97.4 from Rowe et al. (2005) [3], 41 and 135 from Ganne et al. (2008) [26].

When the Freundlich model is applied, we find  $K_f$  and 1/n values ranging from 7.62 to 97.53  $\mu g^{1-1/n} m l^{1/n} g^{-1}$  and from 1.28 to 1.74 respectively. In this case, it is observed that there is a considerable difference between the woven geotextiles and the needled geotextiles for certain chlorophenols. It should be noted that the coefficients of determination show that the adjustment of Freundlich is the most reliable. Indeed, the evolution of the linear partition coefficients with the contaminants always has the same tendency for the two geotextiles, it is not obvious for the Freundlich settings. It should also be noted that pentachlorophenol has the highest adsorption tendency *vis-a-vis* the other pollutants studied.

An important observation is that the amount of chlorophenols sorbed on geotextiles increases with an increasing number of chlorine atoms. Figure 2 shows the relationship between the partition coefficient (log  $K_{ow}$ ) of chlorophenols and amount sorbed at equilibrium ( $q_e$ ) by the two different geotextiles. In the case of chlorophenols polarity appears to be connected with the differences in adsorption.



Fig. 2. Relationship between the partition coefficient (log  $K_{ow}$ ) of chlorophenols and amount adsorbed at equilibrium by the different geotextiles

#### 4.2. Results of batch adsorption tests for bentonites

The experimental results obtained for the determination of the adsorption parameters concerning the geotextiles are presented in the form of isotherms in Figure 3 for BNT1. Adsorption coefficients of the various chlorophenols on the two different bentonites are given in Table 5.

The values of the partition coefficients  $K_d$  of different phenols for different bentonites are 1.5 to 3.2ml / g and fall well within the range given by Richards and Bouazza (2007) [27] ( $K_d = 1.5$ ml/g) and by Haijian et al. (2009) [30] ( $K_d = 1.2$ -3.3ml/g). We did not notice any difference between natural sodium bentonite and activated calcium bentonite. However, these values remain slightly weak compared to those found previously for geotextiles.

If the Freundlich model is used, we find  $K_f$  and 1/n values ranging from 10.72 to 29.23  $\mu g^{1-1/n} m l^{1/n} g^{-1}$  and from 1.27 to 1.54 respectively. In this case, it is observed that there is some difference between the powdered bentonites and granulated bentonites for some phenolic compounds.

Accordingly to what was observed for geotextiles, the sorption of chlorophenols increases with an increasing number of chlorine atoms, *i.e.* as the number of chlorine atoms on the phenol structure was increased, the hydrophilic features of these molecules decreases, and there solubility in water molecules decreases leading to an increase in the adsorption (Figure 4). This result is consistent with previous results from Tahani et al. (1999) [31] on organo-clays.

	BNT1			BNT2						
	F	reunlic	h	lin	ear	Freunlich			linear	
	K <sub>F</sub>	1/ <i>n</i>	$R^2$	K <sub>d</sub>	$R^2$	K <sub>F</sub>	1/ <i>n</i>	$R^2$	K <sub>d</sub>	$R^2$
2-CP	14.61	1.52	0.99	1.50	0.86	14.51	1.52	0.99	1.50	0.88
4-CP	14.20	1.52	0.99	1.50	0.89	14.51	1.52	0.99	1.50	0.84
2,4-DCP	21.66	1.54	0.99	2.00	0.92	21.91	1.54	0.99	2.00	0.88
2,4,6-TCP	18.70	1.46	0.99	2.30	0.90	18.59	1.46	0.99	2.30	0.89
2,3,4,6-										
TeCP	10.72	1.27	0.99	3.20	0.89	10.75	1.27	0.99	2.50	0.92
2,3,5,6-										
TeCP	24.14	1.53	0.99	2.60	0.90	24.10	1.53	0.99	2.60	0.91
PCP	29.23	1.53	0.99	2.60	0.91	29.15	1.53	0.99	2.60	0.90

Table 5. Synthesis of partitioning coefficients for the bentonites of the chlorophenols under study at 23°C



Fig. 3. Results of batch adsorption tests for BNT1



Fig. 4. Relationship between the partition coefficient (log  $K_{ow}$ ) of chlorophenols and amount adsorbed at equilibrium by the different bentonites.

#### 4.3. Results of Partitioning test for HDPE geomembrane

The values of the  $S_{gf}$  partition coefficients of different phenolic compounds in a geomembrane range from 2.64 to 205.51. The highest  $S_{gf}$  value equal to 205.51 was observed for the PCP compound, followed by the 2,3,5,6-TeCP for which the  $S_{gf}$  is equal to 38,49, the 2,4,6-TCP (Mw = 18.01), 2,4-DCP ( $S_{gf} = 8.56$ ), 2-CP ( $S_{gf} = 6.21$ ). The smallest value of 2.64 was observed for 4-CP. Based on the results of this study, we found that the partition coefficient  $S_{gf}$  increases with increasing log  $K_{ow}$  between 2.29 and 5.85, and decreases with increasing solubility S of 0.014. at 28g/l as shown in Table 6. This same result was also observed by Sangam and Rowe [9] for chlorinated hydrocarbons (dichloromethane, 1,2-dichloroethane and trichloroethylene) and aromatic hydrocarbons (benzene, toluene, ethylbenzene, m-p-xylene and o-xylene). These results are also illustrated in Figure 5 by way of comparison; the  $S_{gf}$  values of the phenolic compounds obtained in this study remain low compared to those of VOCs whose chemical structure is similar, obtained by different authors [6-9, 11, 28-29, 32-33].

On this point it can be concluded that the presence of the OH function of the phenol on the molecule decreases the partition coefficient because the hydroxyl group, hydrophilic (thanks to its hydrogen bonds) tends to make the molecule soluble. Indeed, while the solubility of benzene is only 1.78g/L at 20°C that of phenol is 90g/L.

Contaminants	S <sub>gf</sub> (-)	Solubility at 20°C (g/l )	p <i>K</i> a	Log K <sub>ow</sub>
2-CP	6.21	27	9.37	2.53
4-CP	2.64	28	8.52	2.29
2,4-DCP	8.56	4.5	7.90	3.20
2,4,6-TCP	18.01	0.434	6.00	3.67
2,3,5,6-TeCP	38.49	0.100	5.02	5.02
PCP	205.51	0.014	4.74	5.85

Table 6. Inferred partitioning, calculated permeation coefficients from diffusion tests



#### 5. Conclusion

Results of sorption tests of various chlorophenols (2-chlorophenol, 4-chlorophenol, 2,4-dichlorophenol, 2,4,6-trichlorophenol, 2,3,4,6-tetrachlorophenol, 2,3,5,6-tetrachlorophenol, pentachlorophenol) on constitutive of two GCLs (geotextiles and bentonites) and HDPE geomembrane were presented in this paper.

The results show that the partitioning coefficient is linked to the aqueous solubility and the noctanol/water partitioning coefficient of the contaminant. This latest result is logical as the less polar the contaminant is, the less it is soluble in water. This results in a potential for a larger adsorption on the geotextiles, bentonites and geomembrane. The result obtained is that the sorption isotherms obtained are nonlinear.

As far as the partition coefficient are concerned, two different trends were observed, first for geotextiles which are nonwoven needle punched, and second for geotextiles which are woven. On the contrary no significant differences were observed between powdered bentonites and granular bentonites, nor between a natural sodium bentonite and activated calcium bentonites. This study also showed that contrarily to what was observed for VOCs, the difference in the adsorbed amount of phenolic compounds between the geotextile and bentonite is less pronounced. Similarly, the  $S_{gf}$  values obtained in this study are low compared to those of VOCs whose chemical structure is similar, tending to show that the partitioning coefficient decreases as the hydroxyl group tends to make the molecule more soluble.

An important observation is that taking in the case of chlorophenols polarity appears to explain the differences in adsorption, since the adsorption coefficients of a compound increases with the increasing number of chlorine atoms in a molecule.

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