Optimization of municipal solid waste leaching test procedure: Assessment of the part of hydrosoluble organic compounds

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ABSTRACT

Despite national recycling campaigns, the amount of municipal solid waste (MSW) to be treated remains very important in France with almost 39% of the waste produced going to landfills. Therefore with the increasing concern over sustainable development and energy valorization, it seems essential to optimize current treatment methods and develop new preparation techniques of the waste. Nevertheless an important first step to take into account is to evaluate the waste using a different method than biogas production. In this perspective, the leaching test (LT) could be used as a tool to evaluate the ability of a waste to mobilize organic and mineral compounds. This research aims at optimizing a leaching test protocol mainly adapted to organic waste in order to be used on MSW to assess the fractions of both fast and slow mobilized organic matter. Several leaching tests have thus been implemented, optimized and compared in terms of accessible organic matter in the waste. Results have shown that the test conditions have a great influence on the mobilization of pollutants. The duration of the test affects mainly the quantity and quality of organic molecules extracted. The renewal of the eluent does not properly simulate the conditions of a landfill. The results would be used to assess the performance and the efficiency of new ways of waste pretreatment.

1. Introduction

Municipal solid waste (MSW) production is estimated at 350 kg/year/person in France. In 2006, 26 million tons of MSW were produced and 39% were landfilled [1]. MSW landfilling remains one of the most common waste treatment processes and the final stage of the integrated waste management scheme. Nevertheless, landfills could be sources of toxic pollutants, produced through the leachate and the biogas. Thus, landfills can present a potential environmental threat over several years. Thus in this perspective, the French legislation requires the monitoring and treatment of leachate and biogas produced in landfills. In addition to that, new waste management strategies are being actually imposed at the European level such as the European Directive of April 26, 1999 (99/31/EC). This directive requires the reduction of the amount of biodegradable waste before landfilling. To answer such directives, various procedures could be available among which is waste pretreatment appears; the control of this phase is achieved through

knowledge and characterization of the easily biodegradable organic matter [2].

To sum up, waste characterization is essential whether the objective is to provide information on what could be released from the waste into the leachate during the process of biodegradation or to suggest a waste pretreatment that better fits the waste profile.

In a waste, there are mineral (inorganic) and organic fractions that are of interest and that require characterization. The inorganic fraction comprises major mineral compounds in ionic form (Ca^{2+}, Mg^{2+}, Na^+, K^+, NH_4^+, Fe^{2+}, HCO_3^-, Cl^-, SO_4^{2-}) as well as other compounds such as borates, sulphites. The other fraction is the organic matter (OM). This latter consists of a complex and heterogeneous matrix that can be mobilized in soluble forms through the leaching process. On site, this OM matter is mainly transferred to the leachate during the degradation process. Thus, it undergoes various transformations which make it a relevant indicator of the different stages of degradation and stabilization of the waste [3]. In a landfill, the evolution of the organic matter is relatively complex and highly dependent on the environmental conditions (humidity, temperature, etc.). This degradation always involves the production of carbon dioxide (CO_2) and later mainly methane (CH_4).

Despite this complex structure, organic matter can be classified into three fractions [4]:

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- Simple compounds easily biodegradable and identifiable by specific analyses (carbohydrates, fatty acids, amino acids and alcohols, etc.).
- Humic-like substances, which include humins, humic-like acids and fulvic-like acids, are relatively rich in carboxylic and hydroxylic groups.
- Non humic-like substances generally called hydrophilic acids.

Humic-like acids are characteristic of a high humification stage; they are composed of high aromatic character which has required additional extraction protocols. One technique is adsorption on both ion exchange resins and non ionic macroporous resins that is successfully used in natural media (aquatic or soils) and applicable to leachate [5]. For this study, the organic matter is characterized by fractionation according to the hydrophobic character of the molecules carried out using XAD and DAX resins.

In fact, several methods are available for laboratory tests in order to determine this type of leached pollution: column experiments, batch tests, tank tests or extractions [6,7]. However, depending on the method employed, the results of the experiments are difficult to compare [8,9]. The parameters applied often fail to reflect natural conditions and do not apprehend correctly the potential of degradation of the waste. Leachability depends on a number of physical parameters (homogeneity, particle size, porosity, permeability of the solid phase, contact time between the liquid and solid phases, and temperature), as well as parameters such as the pH value, redox conditions, chemical reaction kinetics and complexation with other constituents [10].

Batch tests are generally appropriate for the investigation of leaching behavior of inorganic compounds [10]. Some studies used batch tests to determine the quantity of pollutants able to be leached by a stabilized waste [11]. These types of tests do not take into account the biological phenomena which can constitute a limiting factor for estimating certain pollutants that evolve during the phases of degradation [12].

In this perspective, leaching test (LT) can thus be used to evaluate the solubilisation of pollutants [12] and the follow-up of the evolution of the organic matter fraction. Test results are mainly used to answer questions such as: How much of the total content of a pollutant can be released in leaching processes? What is the time-dependent release from the material by equilibrium or diffusion controlled leaching? What changes will the material undergo with time by atmospheric impact or by leaching? [13]. In particular, it has been proved that the type of pollutant leached depends on the optimization of the liquid/solid ratio, the agitation, the sowing and the temperature [14].

Nevertheless, this test has often been criticized on its reliability and its ability to reproduce in situ conditions. The standard EN12457/1-4 suggested a liquid/solid (L/S) ratio based on the particle size but neither the contact times nor the frequency of renewal of the extracting solution were provided. This test has been developed mainly for inorganic compounds. It does not take into account the specific characteristics of organic compounds or the consequences of a process of microbiological degradation. In fact, the European legislation calls for this type of test to determine the acceptability of a waste in landfill.

Therefore, this research aims at evaluating mainly the organic matter released during the several leaching tests in order to optimize leaching test protocol. In parallel, mineral compounds are analyzed to confirm that their evolution is not indicator of a state of degradation. Leaching tests were conducted with a reconstituted standard French waste. The analytical follow-up that provides the characterization of the biodegradable and refractory fractions of the organic matter mobilized in the liquid phase is presented. The quantities of pollutants obtained by each leaching protocol are compared and discussed taking into account the conditions applied to each type of test. In a context of waste treatment processes, these results could be used to assess the performance of either anaerobic digesters or landfills of both untreated and pretreated waste.

2. Experimental study

2.1. Waste composition

Reconstituted municipal solid waste (MSW) shredded at 20 mm is used to simulate a typical French waste (Table 1). The composition is based on the results of a national sorting campaign carried out in France [1]. This reconstituted MSW has $33 \pm 3\%$ of mineral content and $67 \pm 3\%$ of organic matter content [1] and has not been degraded before its using for test.

2.2. Leaching test procedure

Leaching tests were carried out with a liquid/solid (L/S) ratio of 10 during different durations with and without liquid renewal. The L/S ratio of 10 is chosen after previous study (not presented here) and is expected to promote appropriate contact between the waste and the eluent [3]. Distilled water is used as the eluent (pH = 5.8; resistivity = 18.2 MΩ cm) to achieve the leaching of the stored pollution. Practically, 100.0 ± 0.1 g of dry reconstituted waste are mixed with 1.00 ± 0.01 L of distilled water. The mixture is agitated on a horizontal shaking system at 150 rpm. All experiments were carried out at ambient temperature (20 ± 1 °C) in 2 L glass bottles.

In total, 3 tests are performed:

(1) Leaching during 24 h corresponding to normalized operating conditions (EN 12457/1-4) completed by a leaching test of 336 h without eluent renewal and with sampling (5 mL) of the liquid phase every 24 h.
(2) Leaching test with eluent renewal every 24 h during 336 h. At each renewal, the volume of distilled water added is equal to that of leachate recovered.
(3) Leaching test during 120 h with liquid and biogas analysis every 24 h without eluent renewal.

In order to have a representative sample in each bottle, an important amount of waste properly shredded and mixed was first prepared from which the smaller samples were taken. Thus, a rigorous quartering is done in order to have a homogeneous sample. The leaching tests were conducted in triplicate.

2.3. Analytical procedure

The liquid fraction is separated from the solid fraction by centrifugation at 10,000 g for 20 min. In order to limit any biological activity before analysis, all solutions are stored in glass bottles at 4 °C and analysis are carried out promptly. Previous research has shown that during storage the COD might show a decrease of approximately 9% in 15 days [15]. Thus, tests were performed within 24 h in triplicate.

Table 1
Composition of municipal solid waste.

Composition	% (dry weight)
Organic waste	15.8
Paper/CARDBOARD	26.9
Plastic	12.7
Glass	19.1
Metal	5.6
Textile	4.9
Other	15.0

2.3.1. Liquid analysis

The liquid phase resulting from the LT is characterized following parameters related to the organic content: chemical oxygen demand (COD), dissolved organic carbon (DOC), biological oxygen demand (BOD) and specific UV absorbance (SUVA index). The SUVA index (the ratio of the UV absorbance to the DOC) is used to estimate the aromatic character of the organic molecules [16].

All analyses of the liquid phase are realized after filtration through a 0.45 μm cellulose nitrate filter. DOC measurements are carried out using a TEKMAR DOHRMANN (model Apollo 9000) carbon analyzer. COD is measured using the closed reflux colorimetric method (NFT 90-101). UV 254 nm is measured using a doubled-beam spectrophotometer (SAFAS mC2) with 1 cm-long quartz cells. BOD has been carried out during 5 days using Oxitops® made by WTW (standard NFT 90-103) with inoculation by treated wastewater. In addition to the conductivity, the major ions (Cl^-, Na^+, K^+, SO_4^{2-}, PO_4^{3-} or NH_4^+) are quantified by ionic chromatography (DIONEX DX-120). Results are presented in cumulated amounts relative to the dry matter (DM) of waste.

2.3.2. Organic matter fractionation

Organic macromolecules are characterized by fractionation according to the hydrophobicity [17]. This fractionation follows the method used for natural water and applied to leachate [17–19]. The fractionation protocol is based on the polarity of the molecules and leads to distinguish between different classes of compounds that are more or less hydrophobic. Because it uses non-ionic resins in series (DAX-8, acrylic nature, slightly polar and XAD-4, styrene-divinylbenzene type, non-polar). The characteristics of these resins do not change during the degradation.

In the first stage of the protocol, the sample is filtered through a 0.45 μm cellulose nitrate filter to eliminate humins which are insoluble at any pH. In the filtrate, organic macromolecules like humic acids, sugars or proteins are precipitated at pH 2 (C*) with a 37% HCl solution. The sample is later filtered through a 0.45 μm membrane to remove the precipitated humic-like acids (C*) and the second filtrate is fractionated according to the hydrophobic character of organic molecules. Two resins are used to separate the remaining organic matter fractions into three distinct families of which the structure is not well defined: hydrophobic-like substances (HPO*), transphilic-like substances (TPH*) and hydrophilic-like substances (HPI*). The HPO* are adsorbed on the DAX-8 resin, then the TPH* are adsorbed on XAD-4 resin and the HPI* remain in the liquid phase. The relative concentration of each fraction was quantified by measuring the DOC. The volumes of resin and sample used are respectively 5 mL and 156 mL and the percolation flow is fixed at 50 mL/h [18].

This method of OM fractionation is thus indicative of the waste transformation by showing the evolution of the different organic fractions remobilized in the leachate. Indeed, the degree of humification is correlated with the state of waste degradation [17].

2.3.3. Gas analysis

During the leaching test (3), gas analysis is carried out in order to estimate any possible biodegradation activity. On a daily basis, the volume of the gas produced is measured by moving the volume of water in a reversed test-tube (precision ± 0.2 mL). The quality of the biogas (N_2, O_2, CO_2 and CH_4) is determined using a gas chromatograph (Girdel Serie 30 with catharometer detection).

3. Results and discussion

3.1. Influence of leaching test duration on the mobilization of compounds

French legislation requires a period of 24 h of leaching for municipal solid waste. However, according to several authors, a period of 24 h is too short because it does not extract a sufficient and representative quantity of compounds initially present in the waste [20]. The duration of the leaching should be long enough to allow the establishment of a chemical equilibrium. Moreover, the influence of reaction kinetics cannot be adequately assessed during this limited test time [10]. Thus, the impact of contact time is determined by varying it from 24 to 336 h in order to determine its influence on the mobilization of organic molecules of the solid phase to the liquid phase (Fig. 1).

At 24 h of leaching approximately 50% of the amount of organic compounds is mobilized into the solution compared to that after 336 h (Fig. 1); respectively 9.4 ± 1.6 g C/kg DM and 22.8 ± 2.3 g O_2/kg DM for DOC and COD. An important quantity of mineral compounds is also leached in 24 h, about 8.3 g/kg DM of total ions. These results are characteristic of a fresh waste [21]. Between 24 and 336 h of leaching, the evolution of both the DOC and the COD is divided into 3 stages. A modeling of the behavior of the COD during the leaching is shown in Fig. 2. During the first 120 h of leaching (part I), the amounts of DOC and COD increase at a rate of 0.11 g O_2/kg DM/h for the COD (Fig. 2). In part II, a chemical equilibrium seems to be established in the solution; the release of both DOC
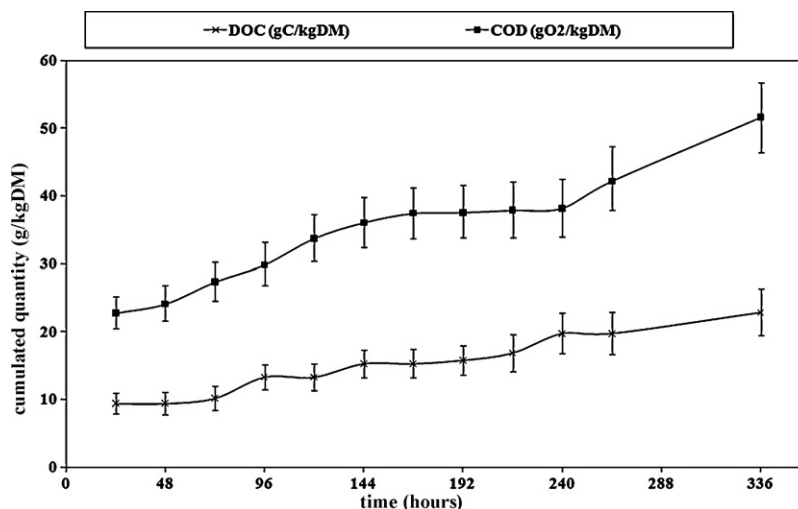

Fig. 1. Experimental results of the evolution of DOC and COD during 336 h of leaching.
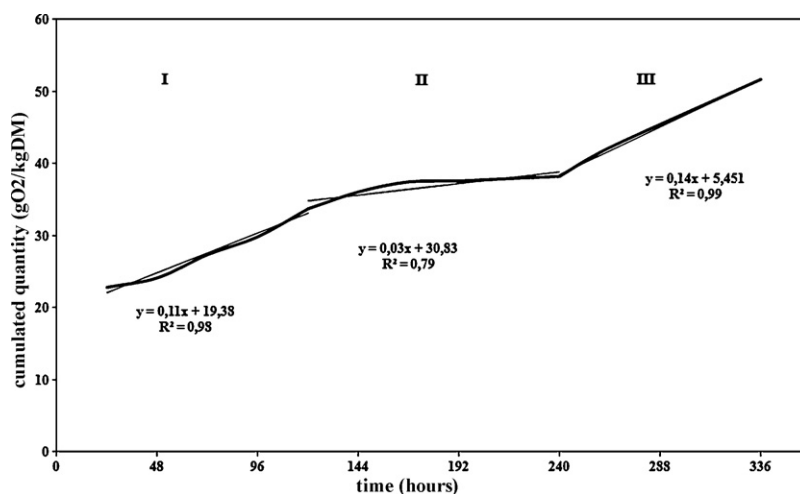
Fig. 2. Equation modeling phases of COD during 336 h of leaching.

Table 2
SUVA index values for leaching test without renewal (L/cm/g C).

Leaching test	24 h	48 h	120 h	240 h	336 h
Without renewal	7.9 ± 0.1	7.7 ± 0.1	7.6 ± 0.1	6.3 ± 0.1	5.0 ± 0.1

and COD is very weak (less than 3 g O_2/kg DM). In fact, a sufficiently long contact time of eluent with waste can lead to an equilibrium between the pollutant content in the solid matter and its concentration in solution; in other words additional mass transfer does not takes place [10]. Nevertheless, this equilibrium can be modified by a biological activity. Beyond 240 h of leaching (part III), a strong mobilization of organic matter is observed. It could be very likely due to the presence of microbial activity in the solution. The type of organic molecules extracted is also influenced by the contact time between the solid and liquid phases (Fig. 3). Results are presented in percentage of each organic fraction.

After 24 h of leaching, the organic matter extracted is still weakly humified. The molecules are mostly hydrophilic. Hydrophilic molecules generally have a low aromaticity which is reflected by a low SUVA index: 7.9 ± 0.1 L/cm/g C (Table 2) [17]. The SUVA index varies between 5 and 10 L/cm/g C for a leachate containing low humified organic compounds but it can reach 100 L/cm/g C for a leachate with mainly hydrophobic compounds [17].

Organic compounds like C* that are highly present after 24 h of leaching are replaced by organic molecules with a less important degree of humification (Fig. 3). This result is confirmed by the decrease in the SUVA index (5.0 ± 0.1 L/cm/g C).

The results obtained after 24 h of leaching provide information on compounds quickly mobilized depending on the composition of the waste. After 120 h of leaching, the transfer from the solid to the liquid phase is weak as both the DOC and the COD do not increase significantly. Thus, an equilibrium state is reached. After 240 h of leaching, the evolution of the above parameters (COD and DOC) reflects the beginning of a biodegradation under anaerobic conditions (Fig. 1). Thus, the presence of methanogenic microorganisms can be underlined by a detection of CH_4 in the biogas produced. Actually, the organic matter has evolved in the solution between 24 and 336 h; compounds have become more hydrophilic. Initial molecules like C* (9.7%) corresponds to easily biodegradable matter like sugars or proteins [17]. Therefore, they have been easily converted into HPO* and HPI* in solution by specific microorganisms.
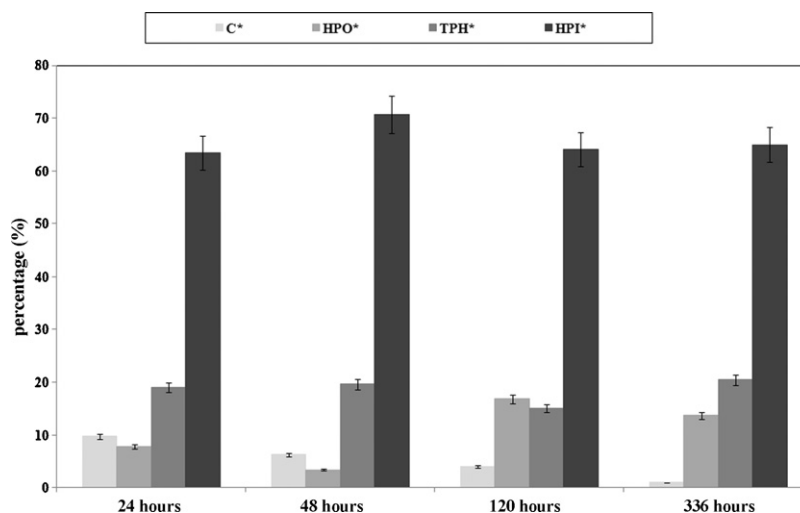

Fig. 3. Fractionation of the organic matter in the liquid phase after leaching without renewal.
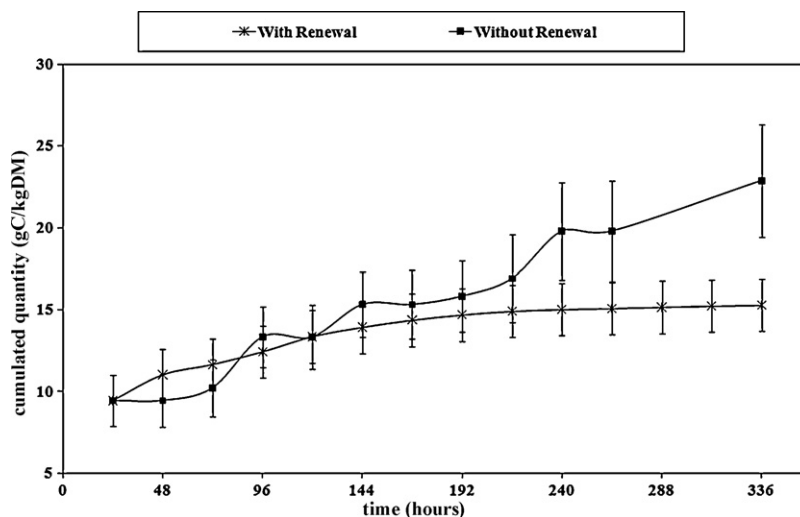
Fig. 4. Evolution of DOC during 336 h of leaching without or with renewal of solution every 24 h.

The amount of mineral species leached increases with the contact time but these mineral species are not indicators of the degradation of the waste because they will not depend on the activity of microorganisms. Their presence in the leachate is mainly due to washing and not to biodegradation [22].

3.2. Impact of the eluent renewal on the mobilization of compounds

Renewing the eluent should provide an estimation of the waste maximal release capacity. The use of distilled water will leach the maximum compounds without saturating the solution. An important amount of oxidizable organic matter is leached during the first 5 renewals (Figs. 4 and 5).

This organic matter corresponds to the quickly and easily mobilized fraction (hydrosoluble). In fact, the residual organic matter remains constant after 14 renewals and represents respectively 15.2 g C/kg DM and 37.9 g O_2/kg DM in terms of DOC and COD. For the same time of leaching, the protocol without renewal can extract more organic matter; respectively 22.9 g C/kg DM and 51.6 g O_2/kg DM in terms of DOC and COD after 336 h of leaching.

Organic compounds like HPI* are released in the first renewals (Fig. 6). These compounds disappear in favor of molecules with a more important degree of humification like TPH* or HPO*. Humic substances like C* are present starting from 24 h of leaching (20.5%). This proportion of C* is a high value for a liquid phase resulting from a leaching of a fresh waste. These molecules are not really humic-like substances but substances which present the same properties by the means of the protocol of extraction [17]. The degradation of organic biopolymer and anthropogenic compounds can generate compounds comparable to humic substances [17,23]. These molecules correspond to easily mobilized compounds like carbohydrates, volatile fatty acids, sugars or proteins [17]. Moreover, the SUVA index increases with the time of leaching reflecting the presence of organic molecules with increasing aromaticity; respectively 8.2 ± 0.2 L/cm/g C and 13.9 ± 1.2 L/cm/g C for 24 and 336 h of leaching (Table 3).

In a leaching test with renewal, only 3–4 days are sufficient to extract a significant amount of organic matter. The repeated leaching solution causes the release of more aromatic organic compounds. The transfer of these organic molecules into the solution is forced by the renewal without complying with the chemical equilibrium of the system.

These conditions of renewals are similar to those obtained in a passive aerobic treatment of waste because waste is in constant contact with oxygen. Without renewal, anaerobic conditions (with-
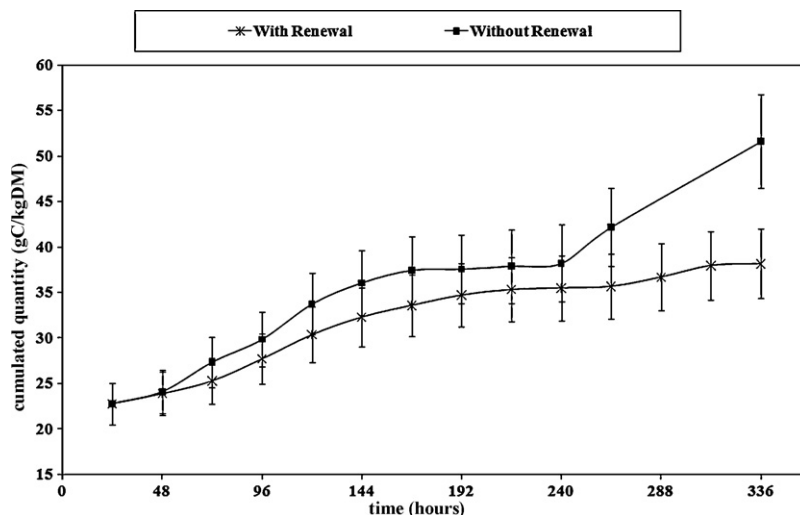

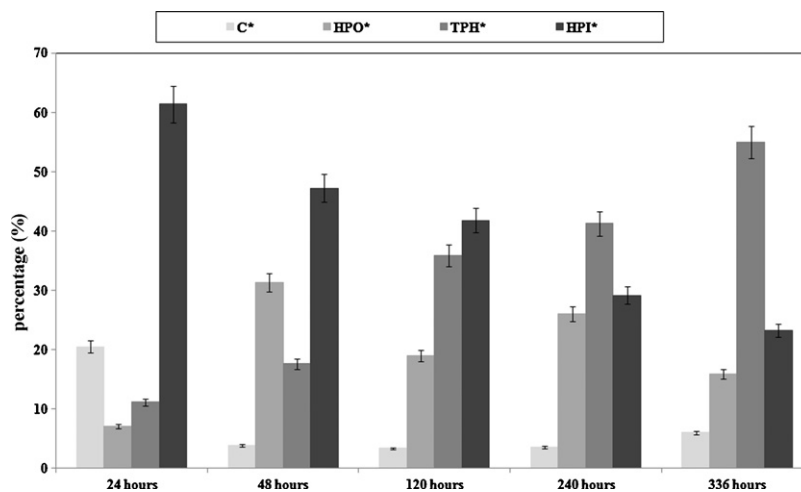**Fig. 5.** Evolution of COD during 336 h of leaching without or with renewal of solution every 24 h.

Fig. 6. Fractionation of the organic matter in the liquid phase after leaching with renewal.

Table 3
SUVA index values for leaching test with renewal (L/cm/g C).

Leaching test	24 h	48 h	120 h	240 h	336 h
With renewal	8.2 ± 0.5	9.8 ± 1.2	10.3 ± 0.1	12.7 ± 1.0	13.9 ± 1.2

out oxygen) are more representative of those of a landfill. Prolonged contact with an unsaturated solution can also simulate the leaching conditions of landfill. The proportion of organic matter mobilized with renewal is weaker than in the test without renewal where there is high biological activity.

3.3. Comparison of leaching test protocol and proposition of an optimal leaching test protocol: 120 h without renewal

This study helps in determining the influence of liquid/solid contact time on the mobilization of molecules from the solid to the liquid phase. The saturation of the eluent is expected with the establishment of an equilibrium state. A compromise between maximum mobilization and minimum biodegradation should be considered. Moreover, in view of the objective of this leaching test, the establishment of a renewal of eluent does not seem necessary.

A period of 120 h of leaching appears suitable for the requirement of minimum degradation. No marked differences between the composition of the organic matter between 24 and 120 h are highlighted. The compounds that are mobilized are good indicators of what is leached. This period of leaching also appears adapted to a maximum mobilization with a low biodegradation. Results show that 21.81 ± 1.19 g C/kg DM of organic matter are extracted in the form of DOC in 120 h of leaching. This organic matter does not correspond to humic-like macromolecules substances; a high proportion of hydrophilic compounds HPI* is leached ($64.1 \pm 1.9\%$).

Another test (without eluent renewal) is performed to validate this period of 120 h. Leaching tests are realized with a leaching time of 24–120 h with measurement of biogas and liquid phases every 24 h. For this test, the evolution of the organic matter reflects the beginning of the biodegradation that was accompanied by a production of biogas under anaerobic conditions (Fig. 7).
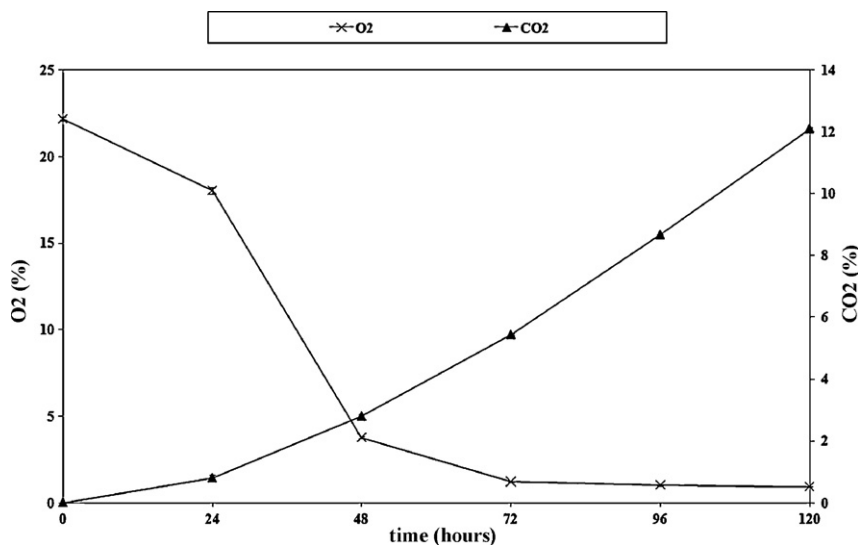

Fig. 7. Evolution of biogas composition in O_2 and CO_2 during 120 h of leaching.
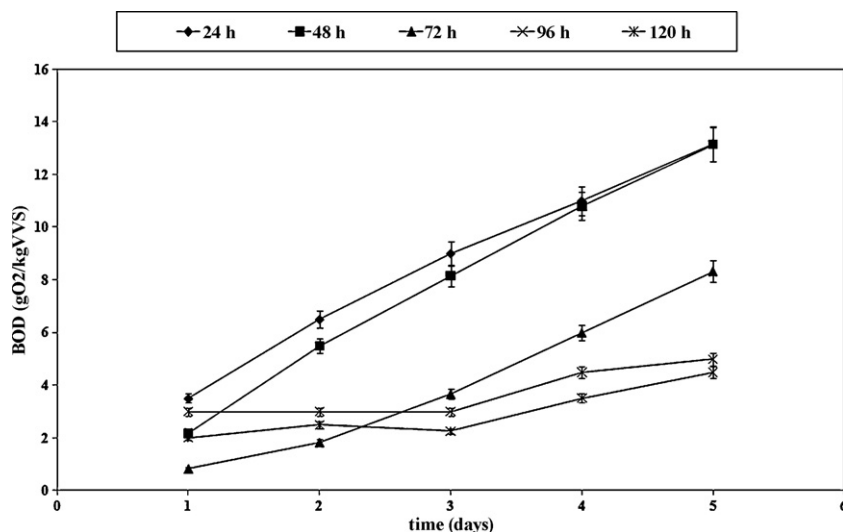
Fig. 8. Evolution of BOD_5 of liquid phase obtained after 24 h, 48 h, 72 h, 96 h and 120 h of leaching test.

Without renewal, the evolution of the gas phase is characterized by a decrease of O_2 content and a production of CO_2 (until $10.11 \pm 0.02\%$) which indicate the establishment of microbial activity under anaerobic conditions. It is important to note that with eluent renewal no biodegradation is observed in the solution. In fact, the eluent is distilled water which stops and/or reduces the biological activity. In addition to that, renewing the liquid phase washes out the microorganisms present in the solution and maintains aerobic conditions because bottles are opened for each renewal. Only the microbial flora attached on the surface of the waste is possibly maintained.

Furthermore, the beginning of this production of biogas in anaerobic conditions is correlated to the decrease of BOD_5 measured in the liquid phase (Fig. 8). The BOD is performed during 5 days on all the liquid phases: after 24 h, 48 h, 72 h, 96 h and 120 h of leaching test. The value of the BOD_5 is used to estimate the biodegradability of the liquid phase and then can determine the biodegradability of compounds leached during the test. The BOD represents the organic matter available to microorganisms. Defining the type of the organic matter (biodegradable or refractory) present is essential to evaluate the environmental impact.

During the first 48 h of leaching, the values of BOD_5 (13.17 ± 0.24 g O_2/kg DM) reflect the presence of biodegradable compounds in the solution. Later BOD_5 decreases (13.15 ± 1.65 to 3.99 ± 0.42 g O_2/kg DM). This evolution of the biodegradability of the organic matter is consistent with the type of organic molecules present in the solution.

4. Conclusion

A leaching test can be used to simulate the leaching phenomenon occurring in landfill. The leaching potential estimated by leaching test depends on several factors related to solid, liquid or experimental procedure applied. Based on the type of the leaching test, the mobilization of the OM, the type of compounds to be quickly dissolved and their ability to be degraded and to produce biogas can be evaluated. The leaching behavior of organic compounds is mainly controlled by dissolution and/or sorption/desorption processes related to the presence of dissolved organic matter [10].

In this study, 3 types of leaching tests were tested. The results have shown that in view of the objective of this leaching test, the establishment of a renewal of eluent does not seem necessary. For a same duration (336 h), the proportion of organic matter mobilized with renewal (15.2 ± 1.6 g C/kg DM) is weaker than in the test without renewal (22.9 ± 3.4 g C/kg DM) where there is high biological activity. The organic matter easily mobilizable (24 h of contact) is hydrophilic (low SUVA index) with an important potential of biodegradation (BOD_5 of 13.17 g O_2/kg DM). Its evolution during leaching indicates the presence of biological activity: consumption of the more easily degradable compounds (decrease of BOD_5).

An optimal leaching test was defined: duration of 120 h in batch with a liquid/solid ratio fixed at 10 can be used to assess the mobilization of organic matter and the type of compounds that can be quickly mobilized. This L/S ratio of 10 is obtained by adding 100.0 ± 0.1 g of dry waste to 1.00 ± 0.01 L of distilled water. The experimental setup is mixed with agitation on a horizontal shaking system at 150 rpm. On a daily basis, determining the quality of the gas produced in terms of O_2 and CO_2 can complete the test and help observe the beginning of the biodegradation. In addition to that, specific parameters like fractionation of organic matter according to the hydrophobicity or evaluation of the SUVA index in the liquid phase provide a typology of fractions readily mobilizable and non-degradable. This test can help compare the behavior of the waste and its ability to be degraded through the species mobilized into the solution with a maximum of mobilization and a minimum of biodegradation. Thus, the protocol defined can allow in a first time to quantify the quantity of pollutant that could be mobilized. The potential degradation of the waste may also be indicated through the kinetics of biological activity.

Acknowledgements

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