Combination of air stripping and biological processes for landfill leachate treatment

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ABSTRACT

Landfill waste decomposition generates a dark effluent named, leachate which is characterized by high organic matter content. To minimize these polluting effects, it becomes necessary to develop an effective landfill leachate treatment process. The objective of this study was to evaluate the performance of an innovative approach based on air stripping, anaerobic digestion (AD) and aerobic activated sludge treatment. A reduction of 80% of ammonia and an increase of carbon to nitrogen ratio to 25 were obtained, which is a suitable ratio for AD. This latter AD was performed in fixed bed reactor with progressive loading rate that reached 2 and 3.2 g COD/L/d for the raw and diluted leachate (1:2), respectively. The anaerobic treatment led to significant removal of chemical oxygen demand (COD) and biogas production, especially for the diluted leachate. The COD removal was of 78% for the raw leachate and a biogas production of 4 L/d with 70% methane content. The use of the diluted leachate led to 81% of COD removal and 7 L/d biogas with 75% methane content. It allowed a removal of 77% COD and more than 97% of the organic compounds present in the initial leachate sample.

Keywords: Activated sludge, Air stripping, Anaerobic digestion, GC-MS, Landfill leachate, Methane production

1. Introduction

Landfill leachate (LFL) is dark effluent resulting from waste decomposition. It contains high amounts of organic matter [1], recalcitrant compounds [2] and emerging contaminants [3]. If it is not properly collected and treated, it may cause a critical environmental problem to groundwater [4]. An effective LFL treatment process is then required to minimize these threats. LFL constitutes one of the most challenging wastewater categories in terms of treatment [5]. In Tunisia, LFL treatment stations adopt an approach consisting of: Coagulation-flocculation, flotation, aerobic treatment, ultra filtration and reverse osmosis, with the aim to meet discharge standards in the natural environment. However, several problems, particularly those related to the effectiveness of leachate treatments are subject to be discussed. They include the poor optimization of the coagulation flocculation process, which may lead sometimes to the elimination of this step, the clogging of the membranes, the dysfunction of the aerobic treatment. Because of these problems, only 1/3 of the leachate is treated and the storage basins are filled with untreated leachate [6]. In addition, this treatment is expensive with a low yield. Simpler process is then required to better suit the nature of the substrate. On the other hand, several studies reported wastewater treatment through physicochemical methods such as photocatalytic degradation [7], adsorption [8] and biosorption [9]. Biological processes are also applied successfully thanks to their efficiency and simplicity [10]. They offer cost effective removal of organic compounds and ammonia nitrogen, leading to a good quality effluent, and preventing pollution transfer [3]. Anaerobic treatment is an efficient biotechnological treatment of highly concentrated organic wastewater. It is one of the most energy-efficient and environmental friendly technologies due to the biogas generation and the low production of sludge [4, 11]. The purposeful utilization of LFL for bioenergy through non-thermal biological methods such as anaerobic digestion (AD) could be a suitable option to tackle the leachate disposal issue. However, there are limited studies on AD for the treatment of LFL for biogas generation [12]. The high organic matter content of Tunisian LFL makes it an attractive substrate for biogas production. Moreover, due to the energy deficit in Tunisia, anaerobic...
treatment presents the best alternative for the leachate treatment. Especially that this treatment is not yet applied in our country. Various anaerobic systems were used for LFL treatment, such as the upflow anaerobic sludge blanket reactors [13, 14], membrane bioreactor [15], sequencing batch reactors [16] and fixed bed reactors [17]. The latter type of reactor attracts more attention following its low sensitivity to toxic compounds, great bio catalytic stability, long microbial residence time and tolerance to oligotrophic conditions [18, 19]. However, the elevated concentration of nitrogen contained in the Tunisian leachate can decrease the anaerobic treatment efficiency by inhibition of microbial activities, accumulation of volatile fatty acids (VFA) and decrease of the methane production [5]. Indeed, Hejnfelt et al. [20] reported that the incidence of ammonia inhibition can happen in the range of 1,500-7,000 mg/L of total ammonia nitrogen. Hence, coupling physico-chemical and biological methods is required for the efficient treatment of LFL. Air stripping, adsorption and coagulation flocculation are the main physicochemical processes applied for leachate pre-treatment [21]. The impact of air stripping process on the methane production by AD of leachate heavily loaded with organic matter was not studied yet. Therefore, the biological approach that combines anaerobic and aerobic processes can be advisable as a handy tool for removing organics from LFL pretreated with air stripping method. Among the main aerobic treatments, we can highlight activated sludge [22], biofilters [23] and rotating biological contactors [24].

In this context, this study investigated the efficiency of the anaerobic treatment of LFL highly charged with organic matter. A pre-treatment using air stripping process was performed in order to reduce the nitrogen load present in leachate and which can constitute a brake on biological development. The high organic load of the Tunisian leachate needs an efficient treatment process to reach the standards of rejects. The chosen treatment is the aerobic treatment using activated sludge system. The objective of this study was to evaluate the performance of this integrated process in terms of organic matter reduction and biogas production. Organic compounds in the raw leachate after anaerobic/aerobic step were measured by gas chromatography coupled to mass spectrometry (GC-MS).

2. Materials and Methods

2.1. LFL and Inoculum

Leachate samples were collected from Sfax landfill site (Tunisia) and stored at 4°C until use. The main physico-chemical characteristics of raw leachate are shown in Table 1. Inoculum used for the AD of leachate pretreated by air stripping was prepared from an existing AD plant for wastewater treatment located in the North of Tunisia (Shotrana) and incubated at 37°C until required for the usage in experiments. Total solids (TS) of 55 g/L, chemical oxygen demand (COD) of 29 g/L and pH of 7.9 characterized anaerobic inoculum. Aerobic sludge comes from the biological basin located in the wastewater treatment plant (WWTP) of Sfax, south Tunisia. It is characterized by a COD of 0.9 g/L, TS of 4 g/L and a pH of 7.5.

2.2. Analytical Methods

The pH, electrical conductivity (EC), TS and volatile solids (VS) were based on the standard methods [25]. COD was determined according to Knechtel [26], biochemical oxygen demand (BOD5) was estimated according to manometric method, total and ammonia-denitrogen were analyzed by Kjeldahl method and VFA were analyzed using a GC-17A gas chromatograph equipped with a capillary column (Nukol: 30 m 0.32 m) and a flame ionization detector.

2.3. Biochemical Methane Potential (BMP) Tests

The effect of air stripping pretreatment on the biogas production was studied by applying the BMP test. The experiments were conducted at 37°C using 500 mL glass bottles as reactors. LFL and acclimated anaerobic sludge were added to bottles, keeping a VS ratio (VS substrate to VS inocula) of 1:1 [27] and the working volume was adjusted to 250 mL. The initial concentration of raw and pretreated leachate are 30 and 28 g/L of COD, respectively. The activity of the inoculum was measured by applying the BMP test. The experiments were conducted at 37°C using 500 mL glass bottles as reactors. LFL and acclimated anaerobic sludge were added to bottles, keeping a VS ratio (VS substrate to VS inocula) of 1:1 [27] and the working volume was adjusted to 250 mL. The initial concentration of raw and pretreated leachate are 30 and 28 g/L of COD, respectively. Based on the initial VS contents of LFL samples (12 g/L) and inocula (20 g/L), the volumes ratio used are 93 and 157 mL for inocula and substrate, respectively. A control batch only with inoculum was used. After adjusting the pH to 7.0, bottles were flushed with N2 for 3 min to supply anaerobic conditions and then incubated in a temperature-controlled room. The measurement of the methane produced was determined using an alkaline solution (NaOH 5% (w/v)) instead water prior the gas displacement device [28].

2.4. Chromatographic Analysis

Samples of raw and treated leachate were collected and analyzed by GC-MS in order to identify the organic compounds. Samples of 500 mL of leachate were prepared according to Ramirez-Sosa et al. [29]. They were extracted three times by 50 mL of dichloromethane, after adjusting the pH to 12 using NaOH 10N. The organic fraction was then recovered in a flask, and the aqueous fraction was subjected again to liquid extraction using dichloromethane after adjusting its pH to 2. The separated acidic and alkaline organic phases were mixed. They were condensed to 5 mL in a rotovaporator and a water bath at 42°C. The condensed fraction was diluted with dichloromethane to 1 mL in an Agilent chromatographic vial and then analyzed by GC-MS using an Agilent 7890A gas chromatograph coupled to an Agilent 7,000 triple quad mass.
of 17 d. was monitored by measuring the COD removal during a period LFL for one time. The ability of the biomass to degrade the OM
The activated sludge system was fed discontinuously with treated
sludge was mixed with treated LFL at a proportion of 1:2 (v/v). A continuous rotation by a stirring system included in the reactor. Aerobic
treatment using activated sludge system was applied as
2.5. Experimental Procedure
The treatment plant was shown in Fig. S1. Experiments were con-
ducted at the laboratory scale.
2.5.1. Air stripping
The experiments were conducted in triplicate at 10 L column with aeration rate of 7 L/min and at room temperature (25 ± 2°C). The pH and reaction time were fixed at 11 and 18 h according to Smaoui et al. [30].
2.5.2. AD of LFL
Upflow anaerobic fixed bed reactor (UAFB) was used for leachate
digestion. It consists of a circular column with 64 cm in height and 17 cm in diameter, offers an effective volume of 7 L and was packed with plastic carriers (type HIFLOWE, cylindrical shape with a specific area of 70 m² m⁻³). The AD was performed at 37°C. Daily biogas produced was measured using a gas meter. The methane content was measured at the end of each organic loading rate (OLR) by using an alkaline solution, prior to the gas meter, which effectively absorbs all the carbon dioxide in the off-gas [28].
To study the OLR impact on the performance of the treatment process, the anaerobic treatment was performed using a raw and
diluted (1:2) LFL.
2.5.3. Aerobic treatment of LFL
Aerobic treatment using activated sludge system was applied as a post treatment. The reactor used had an effective volume of 10 L. An aeration flow of 7 L/min was applied using a diffuser. The temperature was maintained constant at 30°C with a continuous rotation by a stirring system included in the reactor. Aerobic sludge was mixed with treated LFL at a proportion of 1:2 (v/v). The activated sludge system was fed discontinuously with treated LFL for one time. The ability of the biomass to degrade the OM was monitored by measuring the COD removal during a period of 17 d.

3. Results and Discussion
3.1. LFL Characteristics
LFL presents an alkaline pH associated with high EC. This can be justified by the solubility of chloride concentration in solution [31]. The COD content has shown an average value of 30 g/L. The high amount of organic matter makes LFL an attractive substrate for biogas production. The dark color of LFL is explained by the presence of a high content of humic substances [17]. The LFL is characterized by high ammonia concentration (3 g/L) which may cause an inhibition of methanation. In fact, the suitable carbon to nitrogen (C/N) ratio for the methanogenic step is in the range of 20-30 [27]. So the first process applied was the air stripping in order to obtain a C/N ratio suitable for the AD.

3.2. Air Stripping as a Pretreatment of AD
Air stripping was chosen as a pretreatment process to reduce ammo-
nia from LFL and improve the AD. This process is simple and cheaper than other physico-chemical methods with no extra sludge [23]. The application of this process allowed 85% of ammonia reduction and the concentration varied from 2.8 g/L to 0.5 g/L for the untreated and treated leachate, respectively. Following this reduction, the CN ratio was improved to 25 ± 0.7 which is favorable for the anaerobic treatment [32]. Therefore BMP test was performed to ascertain the importance of air stripping in improving methane production during further AD of leachate (Fig. 1). It is clear that ammonia stripping significantly enhanced the methanization of LFL in comparison to raw one. At the end of the experiment, the production of methane increased by 60% for stripped leachate in comparison to the raw leachate. In fact, starting from a raw leachate containing 2.8 g/L ammonia, the addition of a volume of anaerobic sludge inocula led to a dilution of ammonia concentration. However, since the ammonia concentration of sludge is about 0.25 g/L, the final mixture content was about 1.9 g/L. As a consequence, C/N ratio remains low with a value of 3.5 and not suitable for AD which can cause inhibition of methanogenesis. For the bottles containing leachate pretreated by air stripping, the ammonia concentration was about 0.4 g/L and the C/N ratio reach a value of 21 which can be considered as suitable for AD. We can conclude from the batch study that methane production could be due to the degradation of the high strength organic compounds of LFL and decomposition of the non-biodegradable organic macro-
molecules to biodegradable VFA with improved biodegradability [33]. Air stripping contributes to reduce the NH₃ content responsible for leachate toxicity. These results are correlated with those of Liu et al. [34] who indicated that high ammonia concentration can inhibit microbial activities, and so it should be kept below 1,000 mg/L. Chen et al. [35] reported that values of ammonia concen-
tration causing 50% of methane production reduction range from 1.7 to 14 g/L. Yang et al. [36] showed that more than 20% of methane production loss was observed in mesophilic reactors when the ammonia level exceeded 2 g/L. Hobson and Shaw [37] reported that ammonia concentration of 2.5 g/L resulted in some inhibition of methane production, while a concentration of 3 g/L inhibited methanogenesis completely. The study

![Fig. 1. Cumulative methane production during batch anaerobic treatment.](image-url)
reported by el Gohary et al. [38] showed that the use of ammonia stripping improved COD and BOD removal which may improve the leachate biodegradability and improve the volume of biogas released. In addition, Zhang et al. [23] showed an enhanced bio-methanization of piggery wastewater after removal of 80% ammonia by air stripping treatment.

The leachate treated by ammonia stripping is subsequently used in the AD.

3.3. AD of LFL

A UAFB reactor was used for AD experiment. This reactor is the most favored in this case because it makes possible to treat the highly charged effluents while remaining stable even with the variability of the leachate composition and the operating conditions [39]. The decay of organic matter was followed by tracking the soluble COD in the effluent. The reactor was fed with pre-treated LFL. At the first stage, the LFL was used without any dilution. In fact, the high organic matter concentration stimulated the growth of bacteria, that consumed biodegradable organic compounds and then increased the COD removal [40]. During the start-up phase, the anaerobic reactor was discontinuously fed with pre-treated LFL at a low OLR (0.2 g COD/L/d) until acclimatization of the biomass. After exhaustion of the biogas released by the sludge, increasing loads of leachate ranging from 0.5 to 2 g COD/L/d were applied. For these loads, the hydraulic retention time (HRT) decreased from 30 to 14 d (Table 2). The obtained results showed that the COD removal efficiency increases gradually with increasing OLR to reach its maximum of 70% with a load of 1.5 g COD/L/d (Fig. 2(a)). These results are in agreement with other studies which indicated that COD removal increased with increasing organic matter concentration in AD [17, 38]. In general, the efficiency of the AD process is determined by measuring the production of the biogas, which depends on the nature of the substrate to be treated. For this, the daily monitoring of the biogas production shows that it improves gradually with the increase of the organic load applied. A maximum volume of 4 L of biogas is obtained for a load of 1.5 g COD/L/d. The percentage of methane released is between 70% and 80% of the total volume of biogas produced. The continuous production of methane indicates that the methanogenic bacteria degraded the organic matter during the process [41]. The increase in OLR to 2 g COD/L/d reduced the performance of the bioreactor in terms of COD removal and biogas production (Fig. 2) and reach at the end of treatment 72% and 3 L/d, respectively. This can be explained by the destabilization of the process due to the high OLR and to the quality of the influent [42]. In fact, the high salinity of leachate can affect the bacterial population responsible for the degradation of organic matter. In addition, Rahayu et al. [43] explain the decrease of COD removal efficiency by the decrease of the amount of the biodegradable COD. This can also be attributed to the accumulation of volatile fatty acids in the reactor, which have a disruptive effect and cause an imbalance in the process of AD [5, 44].

To study the effect of organic matter concentration on the efficiency of the AD and to improve the performance of the reactor, a diluted LFL (1:2) was used in the next step. Since the reactor was previously acclimated to leachate, it was restarted with an organic load of 1 g COD/L/d and then increased gradually 3.2 g COD/L/d (Table 3). The reactor was fed with LFL having a soluble COD of 15 g/L and the HRT was fixed at 5 d. Under these conditions, the COD removal reached a maximum of 80% (Fig. 3(a)) with...
a production of biogas superior to 7 L/d when the OLR applied was fixed to 3 g COD/L/d (Table 3). The methane content represents 70% of the biogas produced (Fig. 3(b)). These results are very interesting in comparison with other studies. Zayen et al. [17] obtained a 74% of COD removal with only 1 L biogas/d produced, when treating raw leachate with an initial COD of 15 g/L. In addition, Gohary et al. [38] achieved a COD reduction of 41% by applying the anaerobic treatment to treat leachate pretreated by air stripping. The increase of OLR from 3 to 3.2 g COD/L/d, contributed to a decrease of COD removal and a fluctuation in the biogas production, which may be due to dis-equilibrium of the anaerobic process. These data suggest that the use of UAFB reactor can provide a good LFL treatment by reduction of organic matter of 80% and a biogas production of 74%. Indeed, lower LFL treatment performances were described in other studies. For example, Zolfaghari et al. [45] showed a reduction of 63% of COD when using an OLR of 1.2 g COD/L/d in MBR. A reduction of 62% of COD and a methane production of 0.34 L/g COD eliminated were obtained by Xie et al. [46] by treating LFL in MBR.

Despite the interesting results found with AD, the resulting effluent is still not in compliance with the discharge standard allowed by Tunisian environmental legislation especially for the COD (2.8 g/L > 1 g/L). Therefore, it is essential to carry out a post treatment to reduce the concentration of resistant pollutants.

### 3.4. Aerobic Activated Sludge as a Post Treatment

The post-treatment is inevitably applied to guarantee that effluent satisfy the Tunisian strict discharge criteria (NT 106.02). The activated sludge bioreactors were successfully used in conventional wastewater treatment to achieve higher effluent quality [47]. The effluent resulting from the AD with COD of 3 g/L was used as an influent for activated sludge system. The bioreactor containing 3.5 L of activated sludge was fed discontinuously with 6.3 L of treated LFL. The performance of the activated sludge reactor is illustrated in Fig. 4. The results showed that COD removal efficiency increased gradually with time. Therefore, the maximum COD removal was obtained after 17 d of treatment and achieved 77%. Thus, applying activated sludge system as post treatment is very important because it leads to a final effluent that can satisfy the Tunisian limit of discharge.

### 3.5. Determination of Organic Components in Leachate by GC-MS Analysis

An analysis of the organic matter components of raw and treated LFL by anaerobic-aerobic process was performed (Table 4). The compounds present in the leachate samples with spectra signal quality (> 95%) were separated and identified by GC-MS. Fig. S2 shows that the total corrected area of the raw LFL is high compared to the total corrected area of treated LFL. In fact, 26 distinct chromatographic peaks were identified in the raw LFL. The major components identified are phthalates (Phthalic acid, Phthalic acid bis(2-ethyl) ester) and acids (Hexanedioic acid, Heptanedioic acid, Benzeneacetic acid). These components are usually detected in LFL of many countries [29, 48] and known by their toxicities and negative impact on the environment. From the Table 4, we can conclude that the corrected area of LFL treated by anaerobic-aerobic...
| Compounds                                                                 | Raw Leachate | Leachate1 | Leachate2 |
|---------------------------------------------------------------------------|--------------|-----------|-----------|
|                                                                           | Retention    | Corrected | Corrected | % Removal |
|                                                                           | time (min)   | area       | area       |           |
| Hexanedioic acid, bis (trimethylsilyl) ester                              | 4.79         | 128862370  | 33454550   | 74.04     | nd        | 100 |
| Heptanedioic acid, bis (trimethylsilyl) ester                             | 5.45         | 11963341   | 5645722    | 52.80     | nd        | 100 |
| Benzenacetic acid, 3-[(trimethylsilyl)oxy]-, trimethylsilyl ester          | 5.53         | 7385486    | nd         | 100       | nd        | 100 |
| Benzoic acid, 4-[(trimethylsilyl)oxy], trimethylsilyl ester                | 5.69         | 9082462    | nd         | 100       | nd        | 100 |
| Benzenacetic acid, 4-[(trimethylsilyl)oxy]-, trimethylsilyl ester          | 5.77         | 10269156   | nd         | 100       | nd        | 100 |
| 3-Amino-5-cyano-7-ethoxy-2-formyl-4-phenylnithiolo(2,3-b)pyridine          | 5.86         | 9562536    | nd         | 100       | nd        | 100 |
| Octanedioic acid, bis (trimethylsilyl) ester                              | 6.26         | 52437967   | 14675254   | 72        | nd        | 100 |
| Pentanedioic acid, 2,2,3-trimethyl-, bis (trimethylsilyl) ester            | 6.47         | 8676171    | nd         | 100       | nd        | 100 |
| Benzenepropanoic acid, 3-[(trimethylsilyl)oxy]-, trimethylsilyl ester      | 6.61         | 47779300   | 10475555   | 78.05     | nd        | 100 |
| AZELAIC ACID-DITMS                                                        | 7.34         | 16770005   | 41987244   | 74.96     | nd        | 100 |
| 1,2-Benzenedicarboxylic acid, bis(2-methylpropyl) ester                   | 8.48         | 54304721   | 16084391   | 70.38     | 1097347   | 97.97 |
| Sebacic acid, bis(trimethylsilyl) ester                                    | 8.85         | 27990015   | 2885934    | 89.68     | nd        | 100 |
| Benzenedicarboxylic acid, butyl cyclohexyl ester                         | 9.31         | 151807897  | nd         | 100       | nd        | 100 |
| Benzenedicarboxylic acid, bis(2-methylpropyl) ester                      | 9.68         | 51691362   | 32619763   | 36.89     | 30619763  | 40.76 |
| Benzenedicarboxylic acid, dibutyl ester                                  | 10.28        | 103349944  | nd         | 100       | nd        | 100 |
| 1,2-Benzenedicarboxylic acid, dibutyl ester                              | 10.47        | 27747465   | 646875     | 97.66     | nd        | 100 |
| Phthalic acid, butyl isohexyl ester"                                     | 10.75        | 56395469   | 279470     | 99.5      | nd        | 100 |
| 1,3-Benzodioxole-6-carboxamide, N-(2'-benzoylphenyl)                     | 10.95        | 12031538   | nd         | 100       | nd        | 100 |
| Phthalic acid, bis(2-pentyl) ester                                        | 11.27        | 4472031    | nd         | 100       | nd        | 100 |
| Hexadecanoic acid, trimethylsilyl ester                                   | 12.35        | 41484606   | 17095406   | 57.8      | 7447326   | 80.04 |
| Octadecenoic acid (Z)-, methyl ester                                     | 14.11        | 15148482   | 5011653    | 66.91     | nd        | 100 |
| is, 6-Octadeconoic acid, trimethylsilyl ester                             | 19.51        | 15303740   | nd         | 100       | nd        | 100 |
| 2,2-Bis[(4-trimethylsiloxy)phenyl]propane                                  | 19.9         | 24259720   | nd         | 100       | nd        | 100 |
| Octadecanoic acid, trimethylsilyl ester                                   | 21           | 25342317   | nd         | 100       | nd        | 100 |
| Decanoic acid, trimethylsilyl ester                                       | 27.5         | 10131951   | nd         | 100       | nd        | 100 |
| 1,2-Benzenedicarboxylic acid, mono(2-ethylhexyl) ester                    | 30.9         | 131577018  | 79147538   | 39.84     | 77147538  | 41.36 |
| Total corrected area                                                      | 1226757070   | 264145018  | 78.47      | 29166523  | 98        |     |

1. Leachate treated by air stripping-anaerobic digestion
2. Leachate treated by air stripping-anaerobic digestion-activated sludge
nd. Not determined
process decreased 98% in comparison with the corrected area of the raw LFL. The results illustrated an almost complete elimination of the organic compounds present in the raw effluent and thus confirm the effectiveness of applied biological treatments (AD-activated sludge) adopted for the treatment of Tunisian LFL.

4. Conclusions

This study showed that LFL is characterized by a high organic matter, with low biodegradability. It has also a high salts and ammonia content. The air stripping pre-treatment achieved over 80% ammonia removal, which contributed to an increase of the biodegradability and the C/N ratio. The anaerobic treatment was successfully applied for the removal of organic matter, 78% of COD removal for an OLR of 1.5 g COD/L/d and with undiluted LFL. The biogas produced was of 4 L/d. The use of diluted leachate improved the degradation process leading to a COD removal efficiency of 81% for an OLR of 3 g COD/L/d and a HRT of 4 d. The biogas production was 7.2 L/d with 75% of methane content. The application of activated sludge reactor as a post treatment let to remove the resistant organic matter and production of an effluent that satisfies the discharge standard allowed by Tunisian environmental legislation. The GCMS analysis confirmed the effectiveness of the process adopted.

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