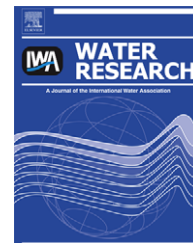


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Continuous treatment of the organic fraction of municipal solid waste in an anaerobic two-stage membrane process with liquid recycle

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ABSTRACT

The stability and performance of a two-stage anaerobic membrane process was investigated at different organic loading rates (OLRs) and Hydraulic Retention Times (HRTs) over 200 days. The Hydrolytic Reactor (HR) was fed with the Organic Fraction of Municipal Solid Waste (OFMSW), while the leachate from the HR was fed continuously to two Submerged Anaerobic Membrane Bioreactors (SAMBR1 and 2). The Total COD (TCOD) of the leachate varied over a wide range, typically between 4000 and 26,000 mg/L while the Soluble COD (SCOD) in the permeate was in the range 400–600 mg/L, achieving a COD removal greater than 90% at a HRT of 1.6–2.3 days in SAMBR1. The operation was not sustainable below this HRT due to a membrane flux limitation at 0.5–0.8 L/m² h (LMH), which was linked to the increasing MLTSS. SCOD in the recycled permeate did not build up indicating a slow degradation of recalcitrants over time. SAMBR2 was run in parallel with SAMBR1 but its permeate was treated aerobically in an Aerobic Membrane Bioreactor (AMBR). The AMBR acted as a COD-polishing and ammonia removal step. About 26% of the recalcitrant SCOD from SAMBR2 could be aerobically degraded in the AMBR. In addition, 97.7 % of the ammonia–nitrogen was converted to nitrate in the AMBR at a maximum nitrogen-loading rate of 0.18 kg NH₄–N/m³ day. GC–MS analysis was performed on the reactor effluents to determine their composition and what compounds were recalcitrant.

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1. Introduction

A major issue in the UK is the shortage of landfills in which to dispose of MSW. In addition, rainwater percolating through landfills leads to the generation of a highly contaminated wastewater (leachate) which is characterized by a high COD and ammonia. Unlike aerobic composting, anaerobic digestion (AD) is an energy producing process that is becoming very attractive due to more restrictive legislation and concerns about carbon footprint. AD of the OFMSW can take place either in dry or wet systems depending on the Total Solids (TS)

content of the reactor. For wet fermentation, the dry matter content is adjusted to 8–16% by addition of process water, whereas for dry systems little or no process water is added to moisten the feedstock. An example of a full scale wet two-stage system is the Schwarting–Uhde process which can sustain an OLR of up to 6 kg VS/m³ day, whereas a full scale dry 2-stage process such as the BRV plant can achieve up to 8 kg VS/m³ day (Trösch and Niemann, 1999). When a biomass retention scheme is added as in the BTA and Biopercolat designs, an OLR up to 15 kg VS/m³ day can be applied successfully (Wellinger et al., 1999; Gallert et al., 2003). The

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biofilm growth in the second stage of the Biopercolat process allows the system to run at an overall retention time of 7 days. In the BTA process the HRT could be reduced to 5.7 days.

For laboratory and pilot scale anaerobic leachate treatment experiments, OLRs from 3 to 22 kg COD/m³ day with COD removal efficiencies of 68–97% and HRTs between 1.5 and 2.6 days have been reported previously (Kennedy et al., 1988; Henry et al., 1987; Chang, 1989). Aerobic leachate treatments, on the other hand, have been applied with removal efficiencies higher than 70% at HRTs ranging from 2.5 to 20 days for leachates with CODs values between 3000 and 48,000 mg/L (Boyle and Ham, 1974; Cook and Foree, 1974; Uloth and Mavinic, 1977; Robinson and Maris, 1985; Maris et al., 1984). However, less sludge is generated and less energy is required if an anaerobic step is followed by an aerobic one. In this process sequence the final aerobic stage serves as post-treatment to improve the final effluent quality (Agdag and Sponza, 2005; Hoilijoki et al., 2000). For instance, Borzacconi et al. (1999) loaded a UASB at an OLR of 20 kg COD/m³ day at an HRT of 2 days and achieved a COD removal greater than 80%; the subsequent aerobic rotating biological contactor achieved 72% COD removal. Another process advantage is the possibility of removing ammonia from the leachate in the aerobic step, but it is known that high influent COD promotes heterotrophic growth and inhibits ammonium oxidation (Cheng and Chen, 1994; Hanaki et al., 1990). Different process configurations have been reported for the simultaneous removal of COD and ammonia from landfill leachate. Im et al. (2001) used an up-flow anaerobic biofilm reactor (36 °C), an aerobic activated sludge reactor (23 °C) and a clarifier achieving an organic removal rate of 15.2 kg COD/m³ day in the anaerobic reactor and an ammonium removal rate of 0.84 kg N/m³ day in the aerobic reactor operating at 4 days HRT. Agdag and Sponza (2005) obtained 98% COD removal of food waste at an OLR of 16 kg COD/m³ day in two UASBs (HRT = 1.25 day) and an aerobic CSTR used in sequence. 99% of NH₄⁺ was removed at 4.5 days HRT in the aerobic CSTR. Chen et al. (2008) used an anaerobic-aerobic moving-bed biofilm system and achieved a COD removal of 92% at an OLR of 15.7 kg COD/m³ day, while 97% of NH₄-N was removed when the HRT of the aerobic step was more than 1.25 days. Jokela et al. (2002) obtained over 90% nitrification at 0.13 kg N/m³ day at 25 °C and 1.4 day HRT in an up-flow filter with crushed bricks.

Another pertinent question related to continuous wet anaerobic fermentation processes when effluent recycle is used is whether recalcitrants such as humic and fulvic acids build up over time, or are slowly degraded. Light metals ions (Na⁺, K⁺, Mg²⁺, Ca²⁺) as well as Cl⁻, PO₄³⁻, SO₄²⁻ and NH₄⁺ may also accumulate to inhibitory levels (Gallert et al., 2003). Leachate recirculation over a tank filled with MSW is relatively well documented (Hao et al., 2008; Bilgili et al., 2007), but recirculation of stabilized leachate in membrane bioreactors is not. Recycling the stabilized leachate to the head of a continuous wet process treating OFMSW could significantly reduce the use of fresh water, and reduce the environmental impact of MSW disposal.

The objectives of this paper were numerous: the effect of the inoculum on the behaviour of the SAMBR was investigated; the stability and performance of the SAMBR was tested at different HRTs and OLRs; and an AMBR operating at

ambient temperature was set up to determine whether the recalcitrants from the SAMBR could be biodegraded aerobically. After 200 days of operation, another objective was to see if there was a build up of recalcitrants with time due to the permeate recycle, or if there was slow degradation, and GC-MS analysis was performed to determine what if any these recalcitrants were. Finally, the different forms of nitrogen were analyzed to determine if nitrification/denitrification was occurring in the system.

2. Materials and methods

2.1. Feedstock

The simulated OFMSW mixture used in this study consisted of 41% Kitchen Waste (KW), 11% Garden Waste (GW) and 48% Paper Waste (PW) on a wet basis. Kitchen waste came from a canteen in Southampton University, UK. The leftovers were passed through a kitchen grinder and mixed in a large tank with a drill mixer and then frozen until the experiment. Garden waste was collected from the Downend Quarry centralised composting site near Fareham (Hampshire, UK) and were kept at 4 °C until the experiment. The composition of the simulated paper waste used for the study is listed in Table 1. This composition comes from an assessment scheme for kerbside collection of dry recyclables undertaken by the district of Eastleigh in the UK. It was deemed a representative paper waste composition because Eastleigh has a good mix of socio-economic groups. Moreover, data of KW, GW and PW are given for summer and winter months, allowing us to see the variation (mainly in garden waste) over the seasons and to calculate an annual average. Finally, Eastleigh has a well established dry recyclables collection of card, newspaper, cans, bottles etc and the data is set out so that actual refuse (Total MSW minus recyclables) is available. This is desirable as legislation says that all city councils in the UK now have to pick up at least two dry recyclables in their waste collections, meaning that Eastleigh refuse MSW is representative of future MSW in the UK.

The TS content of the mixture of waste was adjusted with process liquid to obtain the OFMSW feedstock at 10% TS. In other words, to prepare 1 kg of feedstock at 10% TS, about 830 g of process liquid was mixed with 18.6 g of GW, 71 g of KW and 82 g of PW. The organic content of the simulated

Table 1 – Composition of paper waste used in this study.

Type of paper	%
Newspaper	21
Magazine	12
Office paper	8
Card and paper packaging	11
Cardboard	1
Card non packing	0.6
Liquid carton	1.4
Tissue paper	15
Paper plate	15
Toilet paper	15

OFMSW feedstock was in the range 82–86% of dry matter, and the COD/VS ratio was found to be 1.2–1.6 g COD/g of volatile solids. The ultimate biodegradability was analyzed by Owen et al.'s (1979) bioassay method, and a value of 216 mL CH₄ STP/g VS fed was found at an inoculum to substrate ratio of 0.7.

2.2. Reactors

The HR (10 L working volume) was an acrylic cylinder with a stainless steel mesh which followed a concentric arrangement inside the cylinder, and had a grid of 1 mm holes. A stirrer moved inside the mesh allowing two pieces of rubber to rub against the perforated mesh: the speed of the stirrer (Heidolph) was 40 rpm and was operated intermittently (15 min ON–15 min OFF). The HR was fitted with a 50 micron stainless steel macrofilter (Spectrum Laboratories Inc.) on the inside of the stainless steel mesh in order to retain the large partially hydrolyzed particles, and thereby separate the coarse solids from the leachate being fed to the SAMBRs. The HR and SAMBR1 were connected in series: the leachate containing particulates was fed continuously to SAMBR1 and the permeate from SAMBR1 was recycled to the HR in order to maintain the moisture and alkalinity of the system. On day 45, SAMBR2 was fed continuously on leachate in parallel with SAMBR1 in order to compare the effect of inoculum on the start-up of SAMBR. The HR, SAMBR1 and SAMBR2 were maintained at 35 ± 1 °C. The SAMBRs had a working volume of 3 L, and were made of acrylic panels. They contained a standing baffle designed to direct the fluid to the upcomer and downcomer regimes. The biomass was continuously mixed using headspace biogas that was pumped (Charles Austen Pumps, Model B100SEC) through a stainless steel tube diffuser to generate coarse bubbles. The bubbles pushed the sludge flow upward between the membrane module and the reactor wall in the upper section. A more detailed schematic of the SAMBR and a description of the equipment can be found elsewhere (Hu and Stuckey, 2006). The biogas sparging rate was set at 5 L/min (LPM) using a gas flowmeter (2–20 LPM, ColeParmer, USA) to minimize cake formation on the membrane. On day 130 an AMBR operating at ambient temperature (21–22 °C) was started up to treat the permeate of SAMBR2 continuously. The permeate of the AMBR was then

returned to the HR. The two SAMBRs and the AMBR were fitted with a Kubota polyethylene flat sheet membrane with 0.1 m² total surface and a pore size of 0.4 microns. The final flow-sheet can be seen in Fig. 1.

2.3. Inoculation and start-up of reactors

The HR was inoculated with 4 L of biomass from a previous batch test in the HR at a residence time of 50 days. The inoculum was sieved through a 180 micron screen and its TSS and VSS were 2.74 and 2.07 g/L, respectively. The HR was initially loaded with 400 g OFMSW on a dry matter basis (≈ 340 g VS) in order to stimulate the growth of hydrolytic bacteria, and the volume was adjusted to 10 L with tap water containing NaHCO₃ so that the HR was started up at 4000 mg equivalent CaCO₃/L of alkalinity. The HR was then fed with a load of simulated OFMSW once every two days until day 159, and once a day from day 160 onwards. The simulated feedstock (10% TS) was prepared by adding leachate from the HR to the simulated OFMSW in order to blend the mixture and obtain a homogeneous slurry, and also to minimize fresh water consumption. Fresh tap water was only added to the HR to keep a constant working volume.

SAMBR1 was inoculated with 0.5 L of seed from a SAMBR fed on leachate from the same simulated OFMSW at a HRT of 4 days. The volume was adjusted to 3 L with the anaerobic biomedium defined in Owen et al. (1979) so that the initial MLTSS and MLVSS were 3.31 and 2.54 g/L, respectively. SAMBR2 was inoculated with biomass from a 4 L chemostat batch-fed (once a week) on a 8 g COD/L feed consisting of peptone and meat extract (25% on a COD basis) and a synthetic VFAs mixture (75% on a COD basis) (MLTSS = 1.05 g/L, MLVSS = 0.83 g/L). The concentration of the buffer medium and trace elements can be found elsewhere (Nachaiyasit and Stuckey, 1995). The ratios of the VFAs compared to acetic acid were 1.2, 0.05, 0.22, 0.08, 0.23 for propionate, iso-butyrate, n-butyrate, iso-valerate, n-valerate, respectively. These ratios were typically observed in the raw leachate obtained in previous tests from the simulated OFMSW. Prior to inoculating SAMBR2, a specific acidogenic activity (SAA) test was conducted on the two different inocula, the one from SAMBR1 and the one from the chemostat

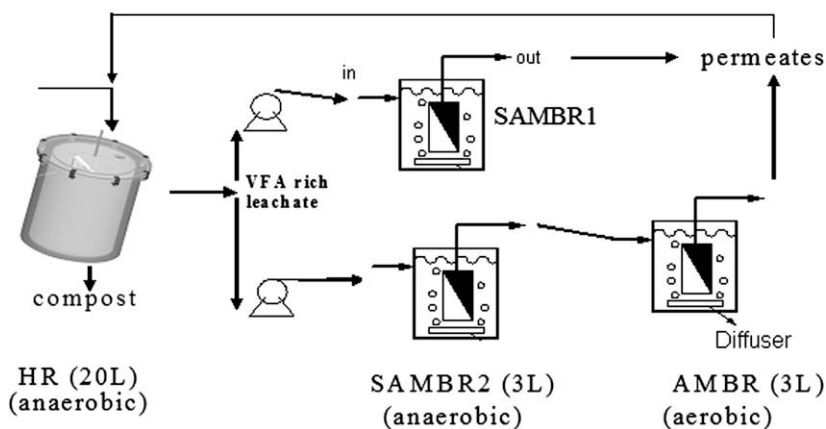


Fig. 1 – Flowsheet of the novel process treating the OFMSW. The permeates from the SAMBR1 and AMBR were recycled to the HR.

batch-fed with meat extract, peptone and synthetic VFAs. The test was determined in triplicate in 38 mL serum bottles, with 20 mL of media. The same amount of glucose was fed to both sets of bottles to result in 2 g COD/L for the test at an initial I/S ratio of 1. Both sets of bottles were inoculated with the same amount of inoculum on a MLVSS basis.

After the SAA test, the supernatant of the chemostat was discarded and the settled solids were used to inoculate SAMBR2. The volume was adjusted to 3 L with an anaerobic biomedium (Owen et al., 1979) so that the initial MLTSS and MLVSS in SAMBR2 were 2.56 and 1.78 g/L, respectively. The AMBR was inoculated with an aerobic biomass from a dye wastewater plant at an initial MLTSS and MLVSS of 3 and 2.3 g/L, respectively. Air was used to mix the reactor content at 1.4 LPM.

2.4. Analytical methods

The measurement of pH (Jenway) was accurate to within ± 0.02 units. The Total Suspended Solids (TSS), Volatile Suspended Solids (VSS), Fixed Suspended Solids (FSS), Soluble Chemical Oxygen Demand (SCOD) and Total Chemical Oxygen Demand (TCOD) were measured as described in Standard Methods (APHA, 1999). Their coefficient of variation (COV) for ten identical samples was 4%, 3.1%, 7.1%, 2.6% and 9.9%, respectively. Volatile fatty acids (VFAs) were measured using a Shimadzu Gas Chromatograph with a flame-ionized detector and a SGE capillary column (12 m \times 53 mm ID-BP21 0.5 μ m). The COV was 3% for ten identical samples. The biogas production rate was measured using the water displacement method; the water was acidified to 2% H₂SO₄ and saturated to 10% NaCl. The composition of biogas was determined using a Shimadzu GC-TCD fitted with a Porapak N column (1500 \times 6.35 mm). The COV for 10 identical samples was 2%. Ammonia–Nitrogen was measured using the Nesslerization method by reading absorbance at 425 nm. The COV was equal to 6.6% for 10 identical samples. Nitrite and nitrate were analyzed by Dionex Ion Chromatography. The COV for 5 identical samples was 1.8%.

For GC–MS analysis, the hydrophobic organic pollutants were extracted using a solid phase extraction (SPE) procedure. The Oasis HLB cartridge (Waters Corporation) was first conditioned with 3 mL methyl tertiary-butyl ether (MTBE), 3 mL methanol and 3 mL deionized water (DW). A sample (500 mL) at pH2 was then loaded onto the cartridge and filtered dropwise. The cartridge was then washed with 3 mL of 40% methanol in DW to remove organic interferences, re-equilibrated with 3 mL DW, washed with 3 mL 10% methanol/2% NH₄OH to remove humic interferences and finally 6 mL 10% methanol/90% MTBE. The final matrix was then evaporated to 200 μ L. The samples were then analyzed using a 5890 Series gas chromatograph equipped with an autosampler and a 5970 mass spectrometry detector (Hewlett–Packard, USA). The analytes were separated using a SGE HT5 column of 25 m \times 0.22 mm with a film thickness of 0.1 μ m. The temperature program was: 50 °C, hold 2 min, rate 8 °C min^{−1}–350 °C, hold 30 s. Helium was used as a carrier gas at a flowrate of 2 mL/min. The injector temperature was set at 270 °C. The MS was operated in the electron impact ionisation mode (70 eV). The transfer line and ion source temperatures were 290 °C and

220 °C, respectively, and the quadruple was not heated. Scan runs were made with a range from *m/z* 33 to 500. The chromatograms were analyzed using the NIST05 library and the compound was deemed identified if the match percentage was higher than 70%. A retention index (RI) was attributed to the unidentified peaks according to Van den Doole and Kratz (1963):

$$RI = 100 \left[z + \frac{t_i - t_z}{t_{z+1} - t_z} \right]$$

where *t_z* and *t_{z+1}* are retention times of the reference *n*-alkane hydrocarbons eluting immediately before and after chemical compound *i*.

3. Results and discussion

3.1. Performance of the hydrolytic reactor

The TCOD in the leachate varied over a wide range, between 4000 and 26,000 mg/L, due to the HR being fed every two days until day 159, intermittent mixing, and due to occasional stirring difficulties. It can be seen from Fig. 2, panel A that the TCOD varies significantly for a given OLR, and this depends on the occasional presence of solid particles in the sampling line at the time of sampling; however, it seems to increase with increasing OLR over the 200 days. Similarly, the SCOD (Fig. 2, panel B) seems to increase with increasing OLRs, but was always in the range 530–2900 mg/L. The evolution and composition of VFAs over time in Fig. 3 shows that acetate was the main acid when no shock was applied, but propionate temporarily became the main acid a few days after the shock at 4, 8 and 16 g VS/L day on days 101, 146 and 164, respectively. From day 160 onwards, the HR was fed on an every day basis at 16 g VS/L day at an HRT averaging 2.2 days, and propionate remained the main acid until the end of the run. Gallert et al. (2003) observed a higher and longer-lasting propionate accumulation when the HRT was reduced from 7.1 days to 5.7 days at an OLR of 15 kg COD/m³ day. They correlated this with 1% hydrogen in the off-gas. Propionate oxidation is known to be the bottleneck reaction during the methanogenesis of complex substrates because the organism carrying out this reaction has a growth rate of only 0.13 d^{−1} (Wallrabenstein et al., 1995). Fig. 2, Panel B shows that the pH dropped to between 6 and 6.5 due to the OLR of 16 g VS/L day applied after day 159, but with the accumulated alkalinity (5000 mg equivalent CaCO₃/L on day 199) and the recycling of the SAMBR1 and AMBR permeates, the pH did not drop any further, which highlights the advantage of recycling the treated permeate which also reduces water consumption.

The low SCOD observed in the leachate was thought to be due to a poor hydrolysis because of the inadequate amount of inoculum used to seed the HR. The initial inoculum to substrate ratio was 0.02 based on the initial load of 340 g volatile solids fed during start-up. Then the HR was fed continuously at an OLR of 0.5 g VS/L day but with intermittent mixing as well as occasional stirring difficulties at TS above 5 %. Table 2 presents the VS removal percentages at the various

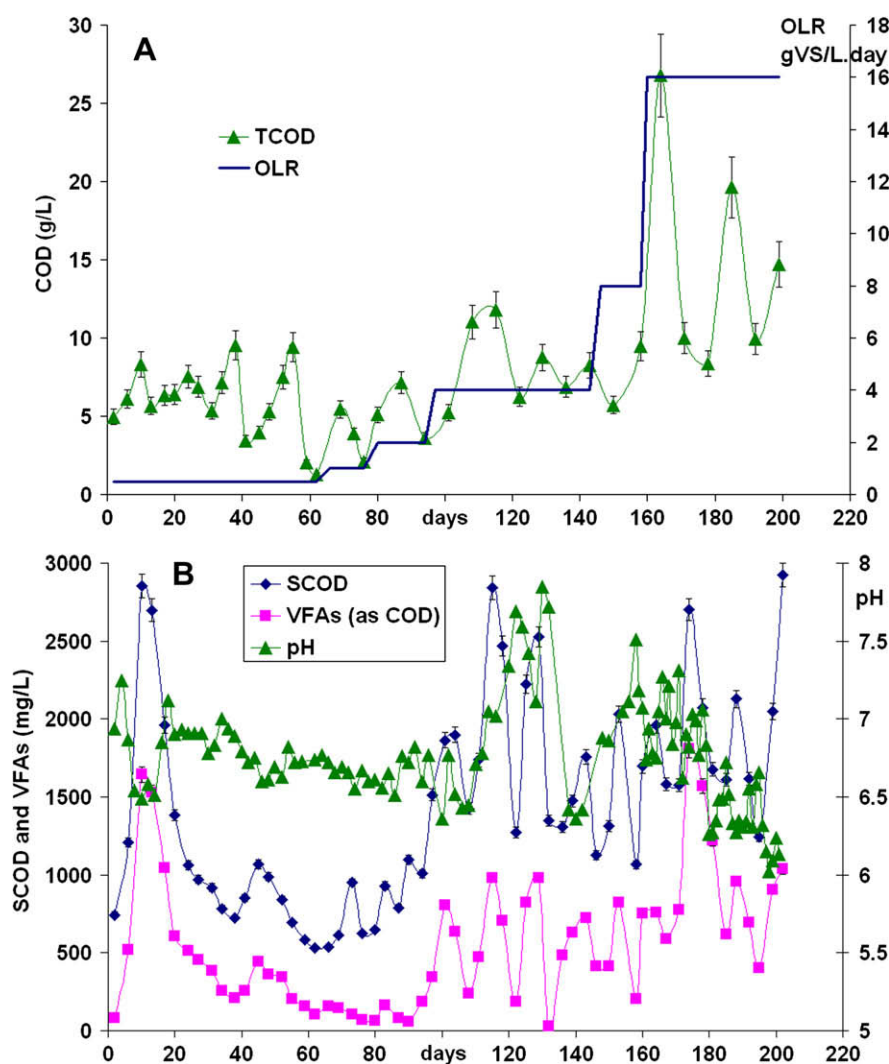


Fig. 2 – A. Evolution with time of TCOD in the effluent of the HR and the OLR to the HR. B. Evolution with time of SCOD, VFAS in the effluent of the HR and pH in the HR (right). The error bars show the standard deviation.

OLRs and HRTs tested. The VS removal % was calculated as follows:

$$\text{VS removal \%} = 100\% \cdot \left(1 - \frac{\text{mass VS removed} + \text{mass VS accumulated in HR}}{\text{mass VS fed in HR}} \right)$$

Where the masses were considered over a period longer than 15 days so that steady-state can be assumed and the mass of VS accumulated in the HR is the difference between the mass of VS in the HR at the beginning and the end of the period considered. The VS removal percentages shown in Table 2 are 65.4, 43.8, 35.5, 22 and 13.8 % VS destruction at 0.5, 2, 4, 8 and 16 gVS/L day, respectively, assuming that the volatile solids production due to bacterial growth and the transfer of volatile solids to the SAMBR were negligible. The transfer of volatile solids to the SAMBR was very limited thanks to the separation between coarse solids and leachate by the perforated stainless steel mesh within the HR. Nevertheless a small fraction of solids could still pass through and be pumped to the SAMBRs. This fraction over 200 days was estimated as 37.8 and 69.3 g VS

for SAMBR1 and SAMBR2, respectively, which can be considered as negligible. For instance, during the period at 16 gVS/L day (day 159–day 199) the total VS mass transferred to SAMBR1 and SAMBR2 together equaled 91 g changing the VS removal % in the HR to 12.4 instead of 13.8. The former is the actual VS removal in the HR, while the later could be named the “apparent VS removal” and in this study they were similar and thus the difference was neglected.

The low VS removal percentages were also due the low volatile solids retention times calculated as the ratio of mass of volatile solids in the HR that is equal to $X \cdot V$ where X is the VS concentration in g/L and V is the reactor volume in L, and the mass of volatile solids removed per day (W in gVS/day): $\text{VS RT (days)} = X \cdot V / W$ (Cecchi et al., 2003). Consequently, the anaerobic biodegradability of the compost of solid digestate that was taken out of the HR was consistent with the lower VS removal observed as the OLR was increased. The BMP of the digestate was 167.7, 229.7 and 296.6 mL CH_4/gVS fed at OLRs of 0.5, 8 and 16 gVS/L day, respectively.

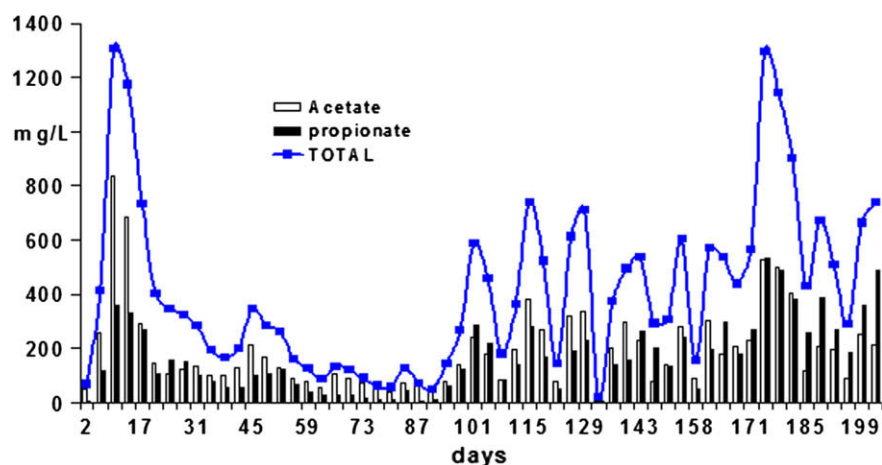


Fig. 3 – VFA distribution in the effluent of the HR.

Table 2 also contains the HRT of the HR, i.e. the hydraulic retention time or leachate retention time, which is the average retention time of a unit volume of liquid in the reactor and is calculated as the ratio of the reactor volume and the leachate flowrate to the SAMBRs. Longer lasting propionate accumulation was observed from day 146 when the HRT was 4 days and also when the HRT dropped to 2 days on day 164. This is in line with Gallert et al. (2003) who stated that propionate oxidizers wash out at HRTs below 8 days.

3.2. Performance of SAMBR1

3.2.1. COD removal

The OLR to the SAMBR was not constant because of fluctuations in the TCOD of the leachate from the HR (Fig. 2), and as a result the SCOD in SAMBR1 (Fig. 4) sometimes increased sharply over time. For instance, an OLR to the SAMBR of 8 g COD/L/day was observed temporarily on day 164, and a simultaneous decrease of the HRT to 2.1 days led to a sharp peak of SCOD in the reactor but this was not due to VFAs building up, indicating that the hydrolysis was rate limiting. On day 185, a maximum OLR of 19.8 g COD/L/day was observed

but the stability of SAMBR1 at such a high OLR could not be assessed due to the transient character of the OLR. Despite the varying OLR, the permeate SCOD (effluent SCOD in Fig. 4) remained typically in the range 300–500 mg/L. A build up of the SCOD was not observed and the SCOD was even found to decrease slightly on a few occasions. This can be partly attributed to the greater consumption of fresh water towards the end of the run to keep up the volume in the HR (see Table 2), but the decline of SCOD was also due to the very high MLTSS (28.7 g/L) at the end of the run, and was not due to the enhanced rejection by the membrane because the SCOD in the bulk liquid was also found to decrease slowly. The SCOD inside the reactor remained higher than the effluent values throughout the experiment, which demonstrates that the presence of a cake/gel layer on the membrane surface considerably improves the effluent quality: this is in line with previous work on the SAMBR (Akram, 2006). Nevertheless, membrane rejection did not increase with time but varied according to the bulk SCOD. Membrane rejection was expressed as a percentage:

$$\text{Rejection} = 100\% \frac{\text{SCOD}_{\text{bulk}} - \text{SCOD}_{\text{permeate}}}{\text{SCOD}_{\text{bulk}}}$$

Table 2 – Comparison of volatile solids removal percentages, fresh water consumption and digestate methane potential at different organic loading rates in the hydrolytic reactor.

OLR (gVS/L day)	0.5	2	4	8	16
Days	0–63	78–95	96–143	144–158	159–199
Duration (days)	63	17	47	14	40
VS RT (days)	67.8	49	16.6	6.4	3.3
VS removal %	65.4	43.8	35.5	22	13.8
HRT (days)	15	9	7.8	4	2.2
Average fresh water consumption (mL/day)	3.7	n.a.	68	202	652
% of fresh water added compared to recycled process water	0.6	n.a.	5	8	14
Digestate methane Potential (mL CH ₄ /g VS)	167.7 ± 6.2	n.a.	n.a.	229.7 ± 6.9	296.6 ± 24
n.a. = not applicable.					

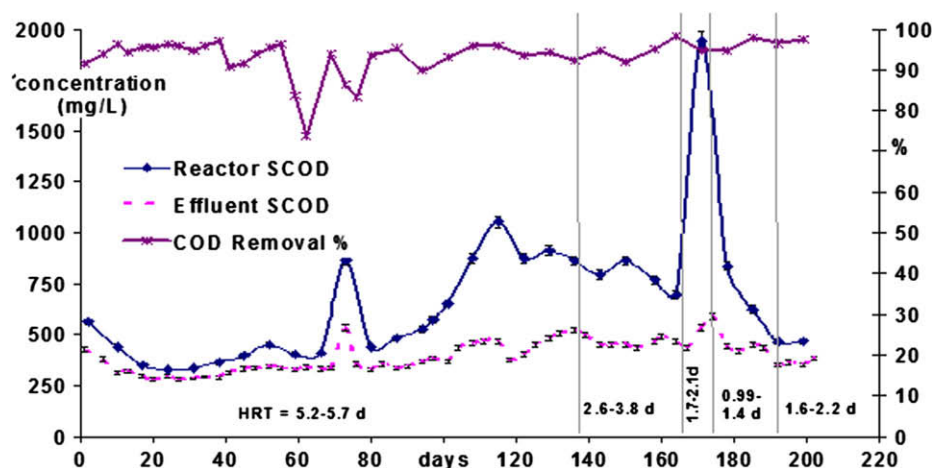


Fig. 4 – SCOD inside SAMBR1 and in its permeate (left axis) at different HRTs. COD removals in SAMBR1 (right axis).

In this study it was observed that the higher the bulk SCOD, the higher the rejection (Fig. 5, Panel A), which suggests that the high molecular weight COD is kept in the reactor and only when it is degraded in the bulk can it pass through the

membrane pores. The COD removal was 93% on average while the total VFA concentration was below 30 mg/L, indicating that the methanogenic population could cope with an HRT as low as 1 day. However, SAMBR1 could not be operated in

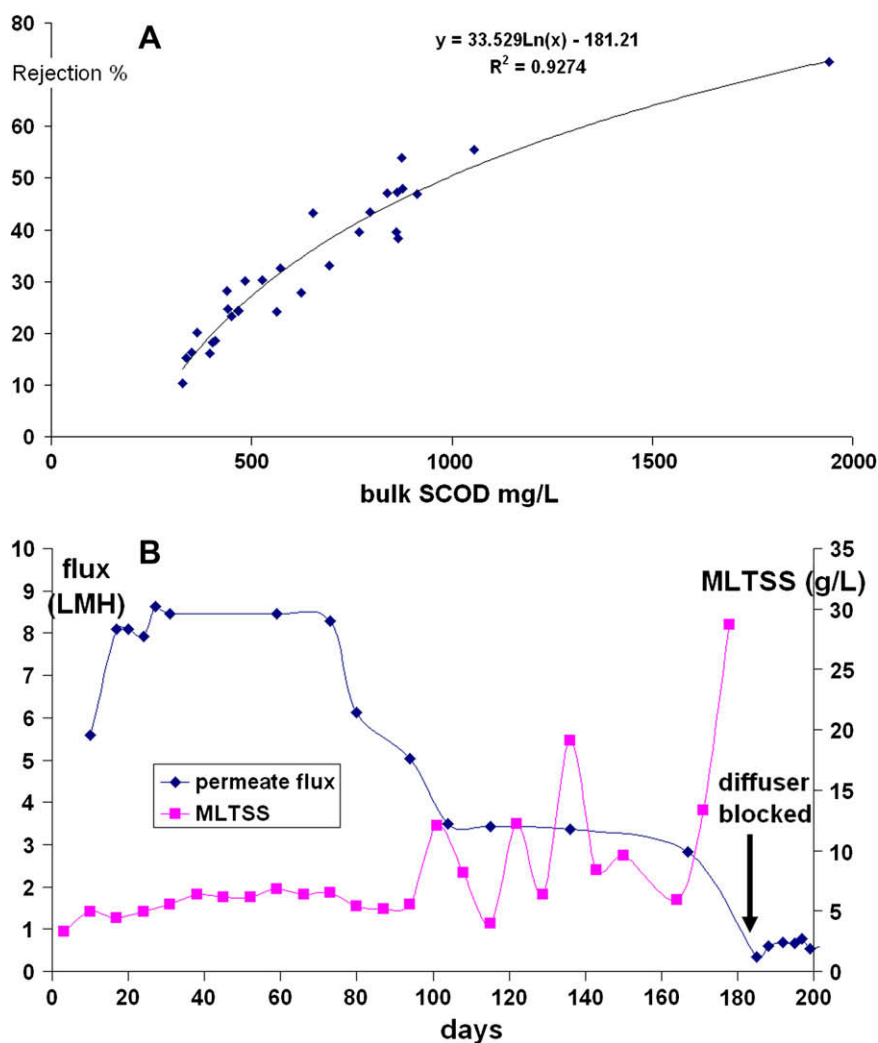


Fig. 5 – A. Correlation between the bulk SCOD in SAMBR1 and the membrane rejection. B. Evolution with time of the MLTSS (right axis) in AMBR1 and the membrane flux (left axis).

a sustainable way at a HRT below 1.6–2.3 days due to a membrane flux limitation of 0.54–0.78 LMH. At an HRT below 2 days, the rate of particulate COD destruction became less than the feeding rate, resulting in the build up of solids at the bottom of the reactor which eventually blocked the diffuser, and on day 182 there were no bubbles scouring the membrane. At the same time, the MLTSS increased to 28.7 g/L (Fig. 5, Panel B) which also adversely affected the flux. This indicates that the performance of the SAMBR treating leachate containing particles was limited to 1.6–2.3 days HRT by particulate hydrolysis and not the VFA degradation.

3.3. Performance of SAMBR2

3.3.1. Effect of inoculum on start-up of SAMBR2

Previous studies (Akram, 2006) have shown that a shorter start-up period and higher COD removal in SAMBRs can be obtained by increasing the organic load at a lower constant HRT rather than gradually decreasing the HRT at constant

high feed strength. This approach was followed to start-up a SAMBR, although Akram (2006) used a sucrose-based wastewater that is easily degradable, while the leachate used in this study was partially refractory. For an easily degradable substrate, VFA accumulation can occur in the SAMBR due to the overloading of the methanogens and possibly the lack of syntrophic associations necessary to degrade reduced intermediates. For this reason, prior inoculation into a CSTR is helpful for the development of an active inoculum enriched in methanogens (Akram, 2006). With this in mind, an inoculum was fed on synthetic VFAs as their main carbon source (75% on a COD basis) in a 4 L chemostat. Prior to inoculating SAMBR2, a specific acidogenic activity test was conducted on the two different inocula, the one from SAMBR1 and the one from the chemostat batch-fed with synthetic VFAs. Fig. 6, Panel A reveals that indeed the acidogenic and methanogenic biomass of the inoculum fed with meat extract, peptone and synthetic VFAs was more active than the inoculum taken from SAMBR1 on a same MLVSS basis. This is due to the large

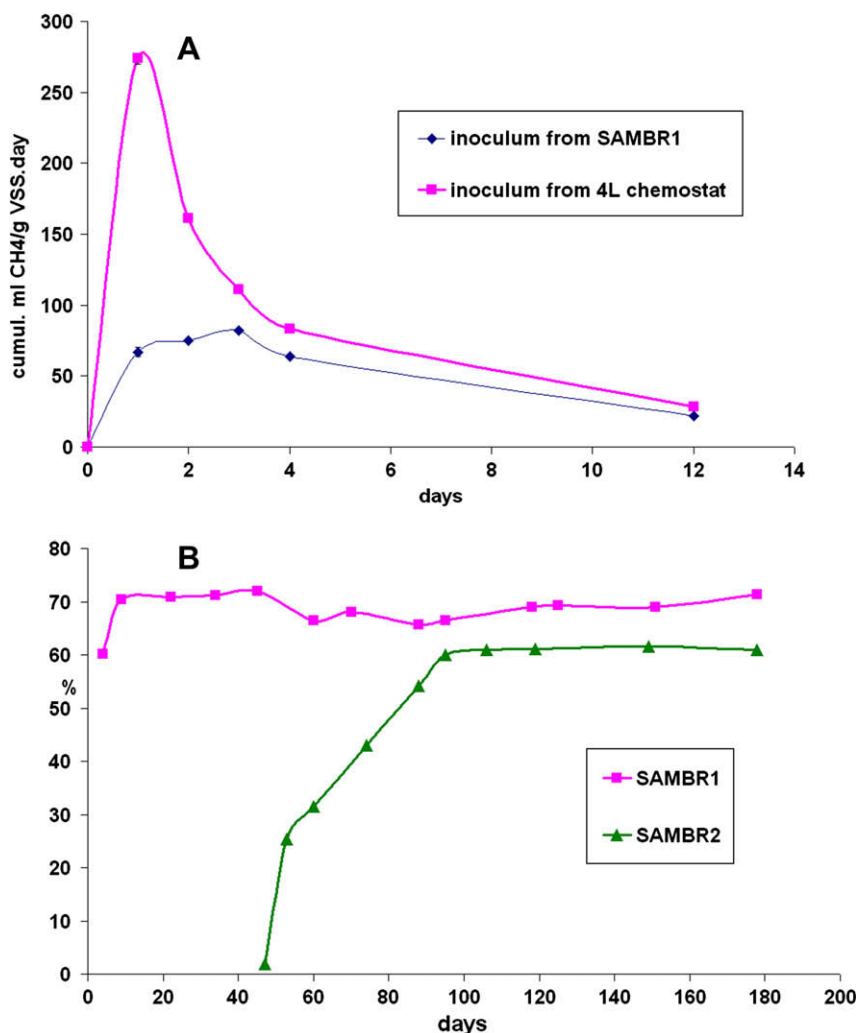


Fig. 6 – A. Specific acidogenic activity test on the inoculum from SAMBR1 acclimatized to the leachate medium and the inoculum from a 4 L chemostat enriched with methanogens in a synthetic medium of peptone, meat extract and VFAs. B. Evolution with time of the methane content of the biogas in SAMBR1 and SAMBR2 inoculation with the inoculum from the 4 L chemostat.

fraction of non-living MLVSS in the inoculum from SAMBR1 that contained lignocellulosic fibers resistant to hydrolysis. As a result, the inoculum from SAMBR1 was likely to contain less acidogens and methanogens explaining the slower methane production rate.

As the acidogenic test showed that the inoculum from a chemostat fed on meat extract, peptone and synthetic VFAs was more active on a MLVSS basis, it was hypothesized that it would be beneficial for the start-up of the SAMBR in order to avoid VFA accumulation. Also less inoculum required to seed the SAMBR due to a higher methanogenic activity would result in lower MLTSS concentrations for a successful start-up.

Total VFA concentrations in SAMBRs1 and 2 were both very low (<30 mg/L). This indicates that an inoculum acclimatized to VFAs such as the one used to start-up SAMBR2 does not bring further advantages regarding VFAs degradation because both SAMBRs at similar initial MLVSS could start-up at a HRT of 5.2–5.7 days with no VFA accumulation. Previous work (O'Sullivan and Burrell, 2007) on leachate from MSW has also shown that microorganisms grown in another medium are unable to out-compete native solid waste microorganisms for cellulose degradation in a foreign (leachate based) medium. In this study, the bacteria fed on peptone and meat extract may have been inhibited when fed suddenly with leachate, or merely may have not been able to metabolize lignocellulosic compounds causing a delay in VFA production and explaining why the methane content displayed such a long lag phase before reaching a normal value of 60–70% CH₄ in the headspace.

Moreover, the methane content of the biogas in SAMBR2 gradually increased to a maximum of 61% after 50 days (Fig. 6, Panel B), whereas in SAMBR1 it reached 60% after four days of operation and then slowly stabilized at values between 69 and 71%, which suggests that the inoculum fed on synthetic VFA was not optimal for start-up because initially it did not contain enough hydrolytic and acidogenic bacteria for a leachate medium. As a result, the slow production of VFAs translated to a slow increase in methane production. However, it could be argued that methane percentage in the headspace is not a good indication of methane production because it depends on the carbon dioxide solubility, which in turn depends on the pH, alkalinity and buffer capacity. In this case, the pH and the alkalinity in SAMBR1 and SAMBR2 were very similar (pH around 7.1 and alkalinity around 2200 mg eq CaCO₃/L – data not shown) which provides a sound base to compare headspace methane content. Furthermore, the bulk SCOD in SAMBR2 kept increasing during start-up from 100 mg/L on day 46 to 800 mg/L on day 73 after which it began to decrease. This strongly suggests that, for a lignocellulosic-based feed, it is paramount to start-up the SAMBR with a competent hydrolytic population acclimatized to the leachate medium to avoid SCOD build up in the bulk. Regarding the gas production rate, no significant biogas was produced in SAMBR2 until day 106, whereas the specific methane production in SAMBR1 was in the range 0.11–0.18 L CH₄/g COD fed.

3.3.2. COD removal

The HRT of the AMBR was equal to the HRT of SAMBR2 because the two reactors were connected in series. The COD removal in SAMBR2 was 94.5% on average, and only 1.6% in

the AMBR so that a total COD removal of 96.1% was achieved. On day 185 and 192, a maximum OLR of 19 g COD/L day was observed with stable COD removal while the HRT was less than 1 day. The total VFA concentration was below 20 mg/L inside SAMBR2 and the permeate, and thus were omitted from Fig. 7, Panel A. No significant change in the contribution to the total COD removal efficiency of both reactors was observed when the HRT was decreased from 5.2–5.7 to 0.37 d. At such a low HRT, particulate solids in the leachate built up at the bottom of the SAMBR2 eventually leading to the diffuser blocking. The MLTSS reached 46 g/L on day 195 (data not shown) which lowered the available flux to 0.4 LMH.

In a moving-bed biofilm reactor system with an anaerobic-aerobic arrangement, Chen et al. (2008) observed that at 1.5 days HRT the COD removal of the anaerobic reactor dropped to 81%, whereas the aerobic COD removal increased to 11%, but nonetheless the total COD of the system remained stable. Although the contribution of the aerobic step to the total COD removal of the system was low in this study (1.6 % on average) because of the membrane rejection in SAMBR2, it should be emphasized that on average 26% of the recalcitrants from SAMBR2 could be degraded aerobically in the AMBR. This percentage was calculated according to:

$$\% = 100 \cdot \frac{\text{SCOD}_{\text{SAMBR2},p} - \text{SCOD}_{\text{AMBR},p}}{\text{SCOD}_{\text{SAMBR2},p}},$$

where the subscript *p* refers to the permeate.

The COD in the permeate of the AMBR was approximately 300 mg/L at the end of the experiment, which is close to the 390 mg/L reported by Agdag and Sponza (2005).

3.3.3. Nitrification in the AMBR

The sequential oxidation of NH₄⁺ to NO₃⁻ involves autotrophic NH₃ oxidizers and autotrophic NO₂⁻ oxidizers. In addition, heterotrophic bacteria can oxidize reduced forms of organic N to NO₃⁻ (Prosser, 2007). Fig. 7, Panel B shows the evolution of inorganic nitrogen species in the AMBR. Because the inoculum used in this study came from a dye wastewater plant, it is assumed that it did not contain any nitrifiers. As a result, ammonia-nitrogen was initially not converted to nitrite or nitrate. Ammonia oxidizers may also have been inhibited by undissociated ammonia (NH₃) which was in the range 14–23 mg NH₃/L between days 136 and 146. Anthonisen et al. (1976) have observed that free ammonia can inhibit ammonia oxidation to nitrite by *Nitrosomonas* and nitrite oxidation to nitrate by *Nitrobacter* in the range 10–150 and 0.1–1 mg NH₃/L, respectively. The nitrite build-up may be explained by the inhibition of nitrite oxidizers due to the free ammonia ranging from 0.1 to 0.4 between days 146 and 167. Inhibition of nitrifying organisms by free nitrous acid (HNO₂) is unlikely to have occurred as the concentration remained in the range 0.00084–0.0052 mg HNO₂/L, which is far below the inhibitory range of 0.22–2.8 mg/L reported by Anthonisen et al. (1976). The growth of *Nitrobacter* was confirmed by the slow decrease in nitrite which was correlated with a slow increase in nitrate. Nitrite was not completely consumed and plateaued at around 60 mg N/L due to HRT shocks. The ammonia-nitrogen in the permeate of SAMBR2 was typically 45–135 mg/L. From day 171 onwards, 97.7% of the NH₄-N was converted in the AMBR at a maximum nitrogen loading rate of 0.18 kg NH₄-N/m³ day.

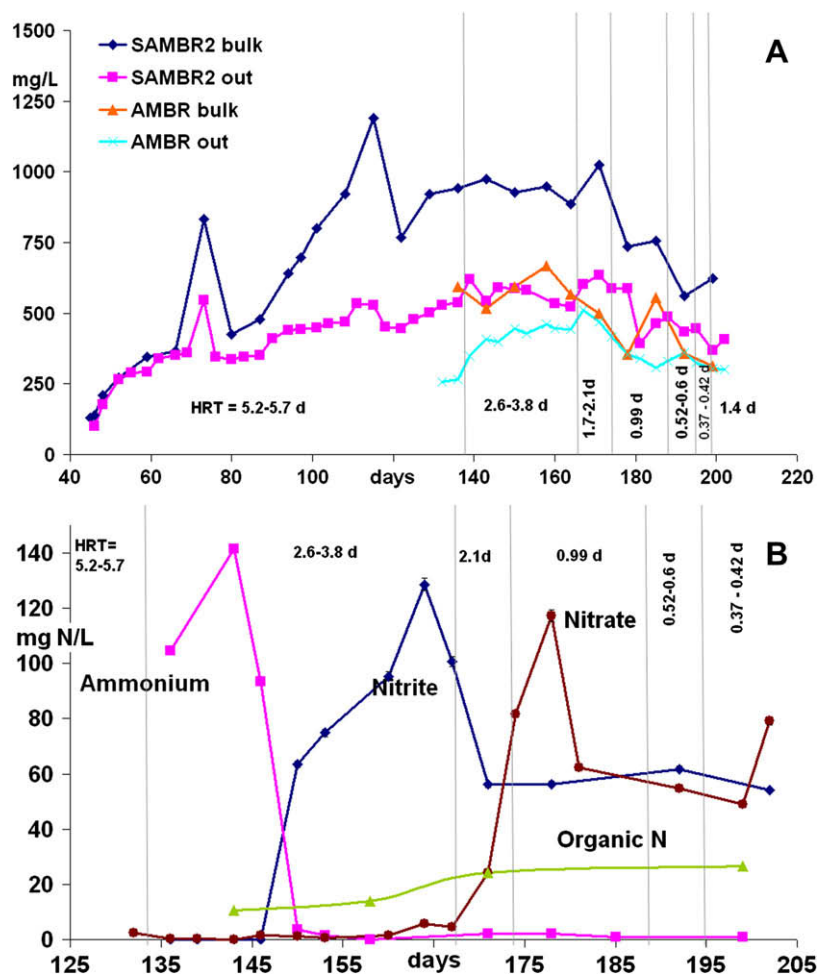


Fig. 7 – A. SCOD inside and in the permeate of SAMBR2 and AMBR at different HRTs. B. Evolution of inorganic nitrogen with time in the AMBR.

The nitrite–nitrate rich permeate was recycled to the HR where denitrification took place because no nitrate was detected in the HR effluent. In this study, the SCOD fed to the AMBR was relatively low (400–600 mg/L) which promoted the growth of autotrophic bacteria. Because of the low organic content and high DO (1.6 mg/L) optimal conditions were met for the growth and retention of autotrophic ammonia oxidizers in the AMBR at a HRT as low as 0.37 day. In contrast, [Chen et al. \(2008\)](#) and [Im et al. \(2001\)](#) could not maintain nitrification at 1.5 and 2.7 days HRT, respectively, because the COD concentration in the feed to the aerobic step increased sharply. [Jokela et al. \(2002\)](#) also observed that nitrification efficiency dropped to below 20% when the COD concentration suddenly increased at 1.4 d HRT. The authors stated that heterotrophs competed for oxygen with the autotrophs leading to a decrease in nitrification activity.

In this study, in addition to ammonia removal in the AMBR, the analysis of Total Nitrogen (TN) revealed that between 7 and 35% of the TN in the permeate of the AMBR was organic N and that organic N was slowly building up in the AMBR. Hence, heterotrophs could very likely have coexisted in the AMBR using organic N for growth and recalcitrant SCOD as a sole carbon source.

3.4. GC–MS analysis

3.4.1. Introduction

The GC–MS analysis performed in this study was qualitative and not quantitative, although comparison between the area of the peaks detected can lead to conclusions regarding the biodegradability in the anaerobic (HR, SAMBR1 and 2) and aerobic (AMBR) reactors. [Tables 3–6](#) document the peaks detected in the effluent of HR, SAMBR1, SAMBR2 and AMBR, respectively, whereas [Table 7](#) lists the retention times of the *n*-alkanes hydrocarbons standard used in the Van den Doole–Kartz retention index. A control sample (referred to as ‘scrap’) consisted of 500 mL of DW in which small pieces of the plastic (plastic scrap) used to make the reactor were added and the mixture was shaken for a few weeks at 30 °C in order to determine which components if any could leach from the reactor’s construction material. [Tables 3–6](#) gather the peak identification number, the match percentage, the retention time, the area and the name of the components that were detected in the effluent of each reactor, but not in the blank (DW that followed the same SPE protocol) nor the control with plastic scraps. In addition, the last column comments on the biodegradability of the compound by comparing the areas of the respective peaks.

Table 3 – Recalcitrants compounds detected by GC–MS in the effluent from the HR. In the case of unidentified compound, the retention index of the peak is given.

peak ID	Percentage match	Retention Time	Reclacitrants in HR effluent	Area
1	93	13.357	butylated hydroxytoluene	12,281,748
2	94	13.72	o-hydroxybiphenyl	16,062,321
3		13.91	1462.7	26,872,283
4	93	16.033	benzophenore	112,030,035
5	93	16.525	tridecanoic acid, 12-methyl, methyl ester	34,198,411
6		17.486	1691.5	56,558,115
7	99	19.141	pentadecanoic acid, 14-methyl, methyl ester	62,748,504
8	99	21.113	8,11-octadecadienoic acid, methyl ester	38,295,039
9		21.271	1964.1	46,078,046
10		21.852	2008.9	95,664,113
11	94	22.481	phenol 4,4'-(1-methylethylidene)bis	570,361,325
12		22.978	2096.1	411,285,760
13	87	24.052	padimate O	101,864,975
14	95	24.39	1-phenanthrene carboxylic acid 1,2,3 m,4,4a,9,10,10a-octahydro-1,4a-dimethyl-7-(1-methylethyl)-,methyl ester,[1R(lalpha,4abeta,10aalpha)]	93,127,843
15	72	26.179	Bis (2-ethylhexyl)phthalate	171,161,815

3.4.2. HR effluent

The analysis revealed that butylated hydroxytoluene ($C_{15}H_{24}O$) and tridecanoic acid, 12-methyl-, methyl ester ($C_{15}H_{30}O_2$) found in the HR effluent were completely degradable because they were not found in both SAMBRs and the AMBR effluents. However, previous work has shown that butylated hydroxytoluene can leach from plastics and tubings (Shpiner, 2007), but it was not detected in our control sample containing plastic scraps. Similarly, 8,11-octadecadienoic acid, methyl ester ($C_{19}H_{34}O_2$) and pentadecanoic acid, 14-methyl, methyl ester ($C_{17}H_{34}O_2$) were two aliphatic molecules that were not detected in the SAMBR permeates due to their complete degradation in this reactor. Surprisingly, padimate O ($C_{17}H_{27}NO_2$) and 1-phenanthrene carboxylic acid 1,2,3,4,4a,9,10,10a-octahydro-1,4a-dimethyl-7-(1-methylethyl)-, methyl ester, [1R(lalpha,4abeta,10aalpha)]- ($C_{21}H_{27}O_3$) that are aromatics and thus considered as difficult to biodegrade were successfully degraded in the SAMBRs due to the complete retention of bacteria. Most of PAHs (polycyclic aromatic hydrocarbons) in

leachate are stable and it is difficult to cleave the ring without oxygen. However, recent research has shown that unsubstituted low molecular weight polycyclic aromatic compounds can be degraded under nitrate-reducing, iron-reducing, sulfate-reducing and methanogenic conditions (Xu et al., 2008).

3.4.3. SAMBR1 and 2 permeates

Tables 3–5 show that o-hydroxybiphenyl ($C_{12}H_{10}O$) and phenol 4,4'-(1-methylethylidene)bis ($C_{15}H_{16}O_2$) (=Bisphenol A) can be considered as non biodegradable because their areas increased by 9 and 12% in SAMBR1, respectively. In SAMBR2 they increased by 123 and 15%, respectively. Several authors have observed that the concentration of Bisphenol A tends to decrease over time in a landfill (Asakura et al., 2004).

On the other hand, Bis (2-ethylhexyl)phthalate ($C_{24}H_{38}O_4$) which is a common plasticizer was not detected in the blank and scrap, and its area more than doubled from the HR effluent to SAMBR1 (+283%) and 2 (+179%) permeates,

Table 4 – Recalcitrants compounds detected by GC–MS in the permeate of SAMBR1. In the case of unidentified compound, the retention index of the peak is given. The last column comments on the anaerobic biodegradability by comparing the peak area of the same compound in the HR effluent.

peak ID	Percentage match	Retention time	Recalcitrants in SAMBR1 permeate	Area	Anaerobic biodegradability
16		11.683	1333.7	26,996,170	New
2	94	13.72	o-hydroxybiphenyl	17,502,568	Non degradable (+9%)
3		13.91	1462.7	14,119,684	Degradable (-48%)
4	83	16.033	benzophenone	21,735,256	Degradable (-81%)
17		18.206	1742.3	15,836,896	new
18	78	18.995	n-phenethylbenznesulfonamide	208,608,266	new
19	78	20.234	2,5-cyclohexadien-1-one,2,6 bis(1,1-dimethylethyl)-4-ethylidene-	44,712,759	new
11	94	22.481	Phenol 4,4'-(1-methylethylidene)bis	640,395,425	Non degradable (+12%)
12		22.978	2096.1	179,375,192	Degradable (-56%)
15	91	26.179	Bis (2-ethylhexyl)phthalate	655,807,845	Non degradable (+283%)
20	95	27.133	Phosphineimide, P,P,P-triphenyl	123,496,720	New

Table 5 – Recalcitrants compounds detected by GC–MS in the permeate of SAMBR2. In the case of unidentified compound, the retention index of the peak is given. The last column comments on the anaerobic biodegradability by comparing the peak area of the same compound in the HR effluent.

Peak ID	Percentage match	Retention time	Recalcitrants in SAMBR2 permeate	Area	Anaerobic biodegradability
21	93	12.4	phenol 2,4-bis(1,1-dimethylethyl)	11,305,194	new
2	94	13.72	o-hydroxybiphenyl	35,846,178	Non degradable (+123%)
22	94	15.697	benzenemethanol, alpha-phenyl	36,535,074	new
4	94	16.033	benzophenone	29,918,768	Degradable (–73%)
18	78	18.995	N-phenethylbenzenesulfonamide	147,543,256	new
19	78	20.234	2,5-cyclohexadien-1-one, 2,6bis(1,1-dimethylethyl)-4-ethylidene-	44,712,759	new
10		21.852	2008.9	46,417,418	Degradable (–50%)
11	95	22.481	phenol 4,4'-(1-methylethylidene)bis	655,737,185	Non degradable (+15%)
12		22.978	2096.1	262,561,636	Degradable (–36%)
15	91	26.179	Bis (2-ethylhexyl)phthalate	478,178,027	Non degradable (+179%)

suggesting that it could be secreted by bacteria themselves, or is the catabolic end product of non detected compounds. Some molecules were found to be slowly biodegradable because their areas decreased when passing through both SAMBRs. These molecules were benzophenone ($C_{13}H_{10}O$) which decreased by 81 and 73% in SAMBR1 and 2, respectively, plus unidentified compounds with retention indices 1462.7 and 2096.1 in SAMBR1 and 2008.9 and 2096.1 in SAMBR2.

3.4.4. SAMBR2 and AMBR

In comparing the SAMBR2 and AMBR permeates it can be seen that phenol 2,4-bis(1,1-dimethylethyl) ($C_{14}H_{22}O$) and N-phenethylbenzenesulfonamide ($C_{14}H_{15}NO_2S$) were not degraded aerobically because their area was found to increase when passing through the AMBR. Mansouri et al. (2007) also found that phenol 2,4-bis(1,1-dimethylethyl) was among the components that remained after an aerobic fixed bed process treating landfill leachate.

Interestingly, some molecules were found to be non biodegradable in an anaerobic environment but could be slowly biodegraded in the AMBR such as 2,5-cyclohexadien-1-one, 2,6 bis(1,1-dimethylethyl)-4-ethylidene- ($C_{16}H_{24}O$) and Bis (2-ethylhexyl)phthalate. The former decreased by 68% whereas the latter decreased by 67% in the AMBR. As Bisphenol A was not found in the AMBR permeate, it can be stated that it was fully biodegraded aerobically. This is in line with Asakura et al. (2004) who treated raw leachate by aeration and found that bisphenol A decreased from 70.9–224 $\mu\text{g/L}$ to 0.11–0.24 $\mu\text{g/L}$ in the effluent. This is also consistent with Spivack et al. (1994)

who found that Bisphenol A is metabolized by a Gram-negative aerobic bacterium via a novel pathway.

Nevertheless, new molecules appeared in the AMBR permeate such as 1,2-benzenedicarboxylic acid, bis(2-methylpropyl)ester and an unidentified peak with a retention index of 1510.6. The molecules 1,2-benzenedicarboxylic acid, bis(2-methylpropyl)ester (= Diisobutyl phthalate) and Bis (2-ethylhexyl)phthalate have a very similar structure with a common ring and two carboxylic groups attached to the ring in ortho and meta positions. Since the area of Bis (2-ethylhexyl)phthalate decreases in AMBR and since 1,2-benzenedicarboxylic acid, bis(2-methylpropyl)ester is a new molecule formed in the AMBR, it is presumed that Bis (2-ethylhexyl)phthalate can lose 2 butyl groups in the two chains attached to the ring to form 1,2-benzenedicarboxylic acid, bis(2-methylpropyl)ester under aerobic conditions which is not possible in an anaerobic environment.

3.4.5. Phthalates and plasticizers

Plasticizers are compounds that are added to polymers in order to improve the properties of a plastic such as increasing its flexibility, and several phthalates were detected in this study. For instance dimethylphthalate was found in the reactor plastic scrap but was not detected in the reactor indicating that it could be readily biodegraded. Diethylphthalate was also found in the scrap but also in the HR effluent and all at a similar abundance of 2,100,000 for the scrap and 2,040,000 for HR effluent. The fact that it was not detected in the SAMBR permeates indicates that it could be biodegraded

Table 6 – Recalcitrants compounds detected by GC–MS in the permeate of the AMBR. In the case of unidentified compound, the retention index of the peak is given. The last column comments on the aerobic biodegradability by comparing the peak area of the same compound in the SAMBR2 permeate.

Peak ID	Percentage match	Retention time	Recalcitrants in AMBR permeate	Area	Aerobic biodegradability
21	91	12.4	phenol 2,4-bis(1,1-dimethylethyl)	16,343,590	Non degradable (+45%)
23		14.7	1510.7		new
18	78	18.995	N-phenethylbenzenesulfonamide	1,307,129,372	Non degradable (+786%)
24	81	20.118	1,2-benzenedicarboxylic acid, butyl (2-methylpropyl)ester	21,287,952	new
19	78	20.234	2,5-cyclohexadien-1-one, 2,6bis (1,1-dimethylethyl)-4-ethylidene-	14,309,919	Degradable (–68%)
15	91	26.179	Bis (2-ethylhexyl)phthalate	156,613,458	Degradable (–67%)

Table 7 – Retention times of standard hydrocarbons detected by the GC–MS used in this study.

Retention time	Hydrocarbons	Formula
6.568	Decane	C ₁₀ H ₂₂
7.256	Undecane	C ₁₁ H ₂₄
9.219	dodecane	C ₁₂ H ₂₆
11.083	tridecane	C ₁₃ H ₂₈
12.866	tetradecane	C ₁₄ H ₃₀
14.532	pentadecane	C ₁₅ H ₃₂
16.109	hexadecane	C ₁₆ H ₃₄
17.614	heptadecane	C ₁₇ H ₃₆
19.014	octadecane	C ₁₈ H ₃₈
20.44	nonadecane	C ₁₉ H ₄₀
21.737	Eicosane	C ₂₀ H ₄₂
23.028	Heneicosane	C ₂₁ H ₄₄
24.16	Docosane	C ₂₂ H ₄₆
25.194	Tricosane	C ₂₃ H ₄₈
26.338	Tetracosane	C ₂₄ H ₅₀
27.337	pentacosane	C ₂₅ H ₅₂
28.355	hexacosane	C ₂₆ H ₅₄
29.33	heptacosane	C ₂₇ H ₅₆
30.267	octacosane	C ₂₈ H ₅₈
31.174	nonacosane	C ₂₉ H ₆₀
32.074	triacontane	C ₃₀ H ₆₂

completely thanks to the long solid retention times achieved in SAMBRs.

Dibutylphthalate was found in the anaerobic reactors but also in the scrap suggesting that it might come from the reactor's plastic. Interestingly, its area decreased greatly in the SAMBRs (from 270 million in HR effluent to 25.6 and 19.9 million in SAMBR1 and 2 permeate, respectively) and was absent in the AMBR, indicating that a great proportion of it can be degraded anaerobically and totally degraded aerobically.

4. Conclusions

The main results of the two-stage membrane process treating the OFMSW are:

- The HR was treating the OFMSW at OLRs ranging from 0.5 to 16 g VS/L day without process instabilities. The main acid in the leachate was acetic acid when no shock was applied, while propionic acid became temporarily dominant when the OLR was increased and was the main acid at 16 g VS/L day. pH drops were avoided due to the permeate stream containing the alkalinity being recycled back to the HR.
- SAMBR1 achieved COD removals greater than 90% at a HRT of 1.6–2.3 days, and recalcitrant SCOD did not build up over 200 days of operation. The permeate of the SAMBR was low in COD thereby providing a stabilized leachate from the very first days of the continuous treatment.
- Inoculation of the SAMBR2 with a bacterial consortium enriched in methanogens in a synthetic biomedium with VFAs as a main carbon source was not optimal for start-up because initially it did not contain hydrolytic and acidogenic bacteria specifically active to treat a leachate medium.
- SAMBR2 achieved COD removals greater than 95% at HRTs as low as 0.4 days. The SCOD permeate was low and constant which did not inhibit autotrophic bacteria in the

AMBR even at such low HRT. The membrane promoted the growth of autotrophic bacteria in the subsequent AMBR so that 97.7% of the NH₄-N was removed at a maximum nitrogen loading rate of 0.18 kg NH₄-N/m³ day.

- GC–MS analysis revealed that the HR effluent contained a number of aliphatic molecules but they were all degraded in the SAMBRs. The permeate of the SAMBRs only contained mainly aromatic recalcitrants molecules, and amongst these, Bis (2-ethylhexyl)phthalate was found to build up in the permeate of SAMBRs but was slowly degraded in the AMBR.

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