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Chemical Engineering Journal 113 (2005) 111–117

Chemical Engineering Journal

www.elsevier.com/locate/cej

Biofiltration of methane: An experimental study

J. Nikiema [a], L. Bibeau [a], J. Lavoie [b], R. Brzezinski [b], J. Vigneux [c], M. Heitz [a,*]

[a] *Faculty of Engineering, Chemical Engineering Department, Université de Sherbrooke, 2500 Boulevard Université, Sherbrooke, Qué., Canada J1K 2R1*

[b] *Faculty of Sciences, Biology Department, Université de Sherbrooke, 2500 Boulevard de l'Université, Sherbrooke, Qué., Canada J1K 2R1*

[c] *GSI Environnement Inc., 855 Rue Pépin, Sherbrooke, Qué., Canada J1L 2P8*

Received 15 December 2004; received in revised form 31 January 2005; accepted 9 April 2005

Abstract

Two filter beds, one composed of inorganic material and the other of mature compost, have been tested for their comparative performance in the biofiltration of methane. The main aim of these tests was to measure the importance of nutrient nitrogen concentration when provided to the bacteria in the form of nitrate ions. The results of this work have revealed that, in a biofilter of 18 L capacity, and fed with 4.2 L/min of gas mixture in which the methane concentration was maintained between 7000 and 7500 ppmv, the inorganic bed proved to be more effective than the organic one. Indeed, the influence of the nitrogen concentration in the nutrient solution is very obvious in the inorganic bed and the optimum level is observed at ∼0.75 g of nitrogen/L, corresponding to a conversion of 41% and an elimination capacity of 29.2 g/m^3/h. With the mature compost-based bed, a maximum conversion of 19% only was obtained, also under a nitrogen concentration of 0.75 g/L. The production of CO_2, the bed temperature and its pressure drop were also examined in this study. The results also show that the inorganic filter bed, in which *Methylocystis parvus* was the main active bacteria, operates at both a higher and a more uniform temperature, and provides for the lowest rate of bed clogging.

Keywords: Biofiltration; Methane; Biogas; Landfill; Methanotroph; *Methylocystis parvus*

1. Introduction

Presently in Canada, methane emissions from landfill sites represent ∼26% of all anthropogenic sources of this greenhouse gas. In the United States, the landfill proportion of overall methane emissions is about 34%. Methane, colourless and odorless, is 21 times more detrimental than carbon dioxide in its greenhouse effect. An appreciable decrease in methane's environmental impact would constitute a worthwhile step towards reduction of the harmful greenhouse gas emissions and the partial achievement of the recommendations of the Kyoto protocol of 1997.

The concept behind the biological treatment of this biogas arises from the fact that some bacteria, mainly the methanotrophs, are able to consume various polluting compounds present in biogas, such as the methane, while only generating water, carbon dioxide, salts and biomass, as the oxidation products, these products being much less harmful for the environment than the initial biogas components. Within landfills, the percentage of the natural biological elimination of the generated methane is estimated to be only ∼10% [1]. The challenge here therefore is to understand the phenomena and processes associated with this biological degradation in order to define the relevant operating conditions that allow for its subsequent application in a biofilter.

1.1. The biogas

Biogas produced by landfill sites is the result of anaerobic degradation of waste. The composition of the biogas and the amounts produced depend mainly on the types of waste stored, as well as the age of the landfill site. The main components of biogas are methane (CH_4), with a concentration ranging from 30 to 70% (v/v) and carbon dioxide (CO_2), with a concentration between 20 and 60% (v/v).

* Corresponding author. Tel.: +1 819 821 8000; fax: +1 819 821 7955.
E-mail address: michele.heitz@usherbrooke.ca (M. Heitz).

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doi:10.1016/j.cej.2005.04.005

Biogas also contains traces of sulfur compounds, such as hydrogen sulfide and some mercaptans and thiols that can be toxic even at low concentrations. These gases are mainly responsible for the unpleasant odors that often emerge from landfills. Biogas also contains small quantities of chlorinated compounds, several of which are toxic and/or carcinogenic. Traces of various other volatile organic compounds (VOCs) such as benzene, which is carcinogenic, as well as toluene and xylenes, are also found in biogas in addition to smaller quantities of hydrogen. Biogas is also generally saturated with water [2,3].

Methanogenic bacteria are mainly responsible for generating the methane found in biogas. In spite of the increasing use of systems for gas emissions collection, only between 40 and 60% (v/v) of the biogas formed is effectively recovered. The residual quantities are dissipated into the surrounding soil and air. Where possible, i.e. when the methane concentration in the biogas is above ∼30% (v/v), the collected biogas can have economic value and practical use. The traditional and most widely used way consists of burning the biogas in boilers for heating and other purposes. Transformation of the biogas methane fraction into methanol can also be carried out [2,4].

When the methane concentration is lower than ∼30% (v/v), collected biogas is burned on the site in order to reduce the risks of explosion associated with the methane content when it is present in air at proportions ranging between 5 and 15% (v/v), but generally no useful heat is recovered [5,6].

1.2. The biofiltration of methane

Experiments conducted in the past by several authors have shown the importance of operational parameters such as the process temperature, pH, moisture, type and texture of the filter bed and the substrate residence time in the biofiltration of methane. The quantity of nutrients available for the bacteria and the presence of inhibitors also influence the behavior of microorganisms in the biofilter [4,7–10].

Many prior experiments have shown that various compost materials constitute adequate filter media for the biofiltration of methane. Indeed, with such composts, reaction times are reduced and overall conversions are often greater and longer than those obtained with other filter bed materials, e.g. soils. Best results are obtained in composts when the organic matter is almost completely stable and the 7-day respiratory activity value is lower than 10 mg O_2/g of dry matter [11].

Both the natural presence of nutrients in composts and their physical properties aid the growth of methanotrophs. The optimal process temperature in composts is 29–30 °C, the optimal moisture content ranging between 25 and 50 wt.%-wet basis [2,5,9]. Soils, particularly those originating from the covers of landfills, can also act as good filtering beds when sufficient nutrients are provided and the moisture levels optimized.

Biofiltration of methane with inorganic materials does not seem to be an attractive alternative. To our present knowledge, only one paper has reported results relative to biofiltration by percolation on glass tubes [12].

The aim of this present study has therefore been to measure the influence of the introduced nitrogen concentration, provided to the bacteria in the form of nitrate ions, on the biofiltration of methane. This has been performed using two similar filter bed installations, an organic one (mature compost) and an inorganic one, by comparing the performance of the two filter packings.

2. Materials and methods

2.1. The biofilter

Two identical biofilters were used for these experiments. Fig. 1 presents a simplified flow sheet of the lab-scale, upflow bioreactor. Each biofilter consisted of a Plexiglas tube, 135 cm in height and a wall thickness of 0.65 cm. The internal diameter of the biofilter was 15 cm. Each biofilter included three identical stages, each stage of 45 cm height. At the base of each section, a perforated plate was placed to support the filter material. The total reactive bed volume was ∼18 L. A tank was placed at the base of the biofilter to perform the task of excess liquid collection, the latter arising during the routine bed watering operations.

The biofilter was fed at its base with a mixture of humidified air and methane, in the desired proportions. This air also contained from 0.67 to 0.72 g/m^3 (that is equivalent to 370–400 ppmv) of carbon dioxide and its humidification was carried out in a separate pre-humidification column. Pure methane was provided by Praxair Inc. (Québec, Canada), contained in a pressurised carboy. The exit port of the biofilter was connected to an evacuation system.

The inlet methane concentration of each biofilter was maintained at 4.6–4.9 g/m^3 (that is equivalent to 7000–7500 ppmv) with a feed rate of the overall gas mixture of 4.2 L/min (or 0.25 m^3/h). This is equivalent to an empty biofilter retention time for the gas of ∼260 s. Two filtering beds were tested in our experiments. Filter bed 1 was made of an inorganic material. Due to an existing confidentiality

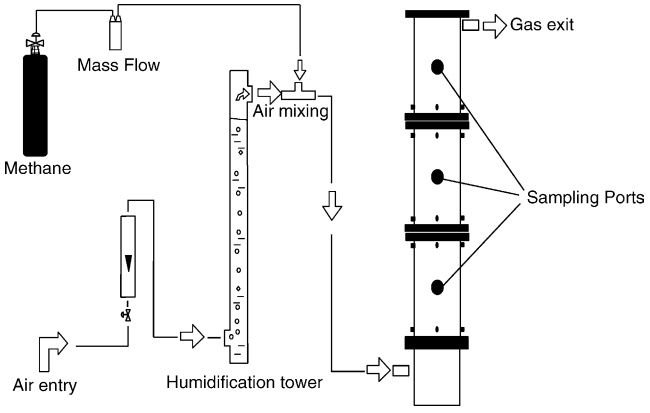

Fig. 1. Experimental lab-scale biofilter.

agreement, specific details about the characteristics of this medium are not available for publication at this time. The filter bed 2 was made of an organic matter consisting of mature compost, sieved using a 6 mm mesh size grid. The compost, used in this study, was provided by GSI env. (Sherbrooke, Qc, Canada). The organic material was not inoculated.

In order to inoculate the inorganic-based bed, 50 g of soil sample from Bessette landfill located near Sherbrooke (Qc, Canada) were mixed with 0.8 L of NMS (nitrate minimal salt) solution and incubated under a 1% (v/v) methane atmosphere at room temperature. After elimination of the coarser particles by low speed centrifugation, the supernanant liquid was used for the inoculation.

2.2. Operating conditions

Filter bed humidification was ensured by daily watering with 1.5 L of a nutrient solution. This solution was prepared with Sherbrooke (Québec, Canada) tap water to which was added various compounds in the desired proportions. The composition of this solution is similar to that of the NMS, as used by Fox et al. [13]. In the nutrient solution, small amounts of iron and various other metal ions, phosphate, mineral salts, and nitrogen as nitrates are present. The nitrogen concentrations were periodically varied, in order to measure the influence of the concentration of nitrogen, the selected concentrations being: 0.14, 0.25, 0.5, 0.75, 1.0 g/L.

2.3. Physical and chemical analysis

The inlet methane concentration and at the exit of each stage were measured daily by means of a flame ionization hydrocarbon analyzer (Horiba model FIA-510), while the carbon dioxide concentration was monitored with a Siemens apparatus (Ultramat 22P). Additional information about the gas analysis is given in the paper by Jorio et al. [14].

The temperature of the gases admitted to the biofilter ranged between 15 and 25 °C throughout the experimental period. The temperature at each stage of the biofilter was also monitored. These measurements were made through the sampling ports, placed in the middle of each stage as presented in Fig. 1, by means of a type T thermocouple, connected to a read-out unit (Omega, model DP465). The pressure drop was also monitored, via a differential manometer (Air Flow Developpement Ltd., UK, type 4). All of the measurement data were received and stored with the aid of data acquisition software.

2.4. Microbiological analysis

In order to identify the dominant microbial groups in the biofilm population, samples (3 g) of the filter material were vortexed for 10 s, then shaken for 30 min with 30 mL aliquots of the extraction buffer (0.1 wt.% sodium pyrophosphate, pH 6.5; 2 wt.% NaCl). This suspension was then centrifuged, initially at low speed ($800 \times g$) for 3 min to sediment the coarse debris and then at higher speed ($4000 \times g$) for 10 min to pellet bacteria. DNA was then extracted from the pelleted bacterial cells by the method of Kirby et al. [15].

For PCR amplification, 1 μg of the resultant DNA was combined with 0.4 μM of primers for amplification of the rRNA 16S genes (primer BSF 8/20 5′-AGAGTTTGATCATGGCTCAG-3′ and primer BSR 1541/20 5′-AAGGAGGTGATCCAGCCGCA-3′), in PCR amplification buffer (Amersham Biosciences), supplemented with 10% (v/v) dimethylsulfoxide and 0.2 μM of each of the deoxyribonucleotide triphosphates in a final volume of 50 μL. The amplification procedure was started by the addition of 2.5 U of Taq DNA polymerase (Amersham Biosciences). The amplification reaction time included some 5 min of initial denaturation at 94 °C, followed by 35 cycles of denaturation (1 min at 94 °C), annealing (1 min at 54 °C) and elongation (1 min at 72 °C). Amplification was terminated with a final elongation of 10 min at 72 °C.

The DNA segments, now amplified by PCR, were ligated into a linear form of pCR 2.1 vector, following a procedure known as TA-cloning (vector and reagents available from Invitrogen Life technologies, Carlsbad, CA). The vector has two *Eco*RI sites flanking the ligation site, allowing for precise excision of inserts.

Transformation and positive clone selection were carried out according to the manufacturer's guidelines. Forty plasmids with inserts were kept for restriction fragment length polymorphism analysis. These plasmids were digested separately with 10 restriction enzymes: *Eco*RI, *Kpn*I, *Hin*dIII, *Sac*I, *Bam*HI, *Xho*I, *Eco*RV, *Not*I, *Xba*I and *Apa*I. Selected inserts (one per polymorphic pattern) were sequenced using an automatic DNA sequencer, LI-COR Global Edition IR^2 system (LI-COR Biosciences Inc., Lincoln, Nebraska, USA) using fluorescent sequencing primers labeled with the fluorophore IRD800 (LI-COR Biosciences Inc.) and the SequiTherm EXCEL II DNA Sequencing Kit-LC (Intersciences, Canada) for DNA strand elongation in vitro.

The rRNA 16S gene sequences were then compared with a bank of known sequences to determine the taxonomic position of the microorganisms from which they originated [16]. This was established by BLAST-N analysis, using the NCBI (National Center for Biotechnology Information) server at the default settings [17].

The results will be presented in terms of:

- IL: inlet load ($g/m^3/h$)

$$\mathrm{IL} = \frac{C_{(CH_4)\mathrm{in}} \times Q}{V} \quad (1)$$

- X: conversion (adimensional)

$$X = \frac{C_{(CH_4)\mathrm{in}} - C_{(CH_4)\mathrm{out}}}{C_{(CH_4)\mathrm{in}}} \quad (2)$$

- EC: elimination capacity ($g/m^3/h$)

$$\mathrm{EC} = \mathrm{IL} \times X \quad (3)$$

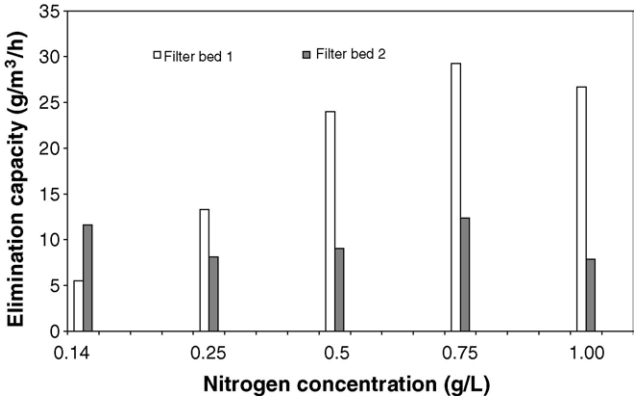

Fig. 2. Elimination capacity for CH_4 (input concentration: 7000–7500 ppmv) expressed in g/m^3/h in an inorganic filter bed (filter bed 1) and a mature compost-based bed (filter bed 2), as a function of nitrogen concentration in the nutrient solution.

- P_{CO_2}: carbon dioxide production (g/m^3/h)

$$P_{CO_2} = \frac{(C_{(CO_2)out} - C_{(CO_2)in}) \times Q}{V} \quad (4)$$

where $C_{(CH_4)}$ is the methane concentration in g/m^3; $C_{(CO_2)}$ the carbon dioxide concentration in g/m^3; Q the gas flow rate in m^3/h and V the bed volume in m^3.

3. Results and discussion

3.1. Elimination of methane

The two biofilters, as used in these experiments, were operated over a period of six months. The quantity of methane eliminated, which depends on the change of methane concentration between the biofilter inlet and outlet, is the key indicator of the effectiveness of the treatment process. The measured elimination capacities for methane, as obtained in both biofilters, are presented in Fig. 2, along with the conversion in Fig. 3.

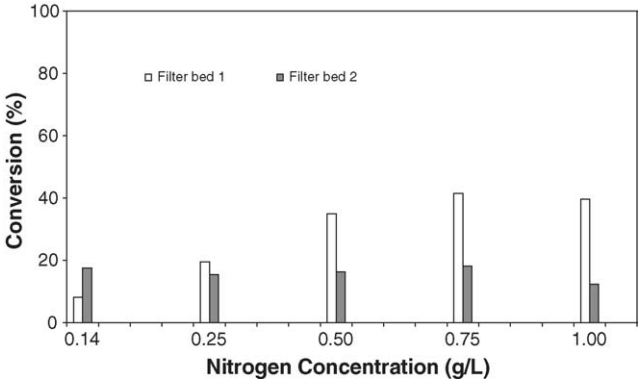

Fig. 3. Conversion values for CH_4 (input concentration: 7000–7500 ppmv) expressed in %, in an inorganic filter bed (filter bed 1) and in a mature compost-based bed (filter bed 2), as a function of nitrogen concentration in the nutrient solution.

The importance of the nitrogen concentration in the nutrient solution for inorganic bed 1 is apparent in Fig. 2. It can be seen that variation in the nitrogen concentration directly influences the elimination capacity. The best results are obtained with an input nitrogen concentration of 0.75 g/L. Under this condition, the elimination capacity reaches 29.2 g/m^3/h and the conversion rises to 41%. When nitrogen concentration in the nutrient solution is raised above 0.75 g/L, biofilter performance decreases. Below the 0.75 g/L value, performance of the inorganic bed material biofilter can be improved to a significant degree. Indeed, each time nitrogen is raised from 0.14 to 0.25 g/L or from 0.25 to 0.75 g/L, methane elimination capacity is more than doubled, i.e. from 5.4 to 13.3 g/m^3/h or from 13.3 to 29.2 g/m^3/h, respectively.

The influence of nutrient solution nitrogen concentration, relative to the biofilter containing the organic filter bed, is not however similar to that observed for the inorganic filter bed. Thus, differences resulting from variations in the nitrogen concentrations of the nutrient solution are not as high as observed in the inorganic filter bed, the elimination capacities varying between 7.9 and 12.5 g/m^3/h. The maximum conversion obtained with the mature compost-based filter bed was ~19%, when the nutrient solution contained 0.75 g of nitrogen/L. This conversion result is approximately only half the value of the maximum conversion obtained using the inorganic material bed, and is similar to that obtained with the 0.14 g nitrogen/L nutrient level (18.5%). The observation can be explained by the fact that the mature compost-based bed possibly contains some intrinsic nitrogen, thereby raising the total concentration of nitrogen available for use by bacteria. Thus the available nitrogen level may be higher than that of the nutrient solution, especially in a freshly prepared filter bed, which corresponded to the period during which experiments at 0.14 g/L nitrogen content level were conducted.

3.2. Carbon dioxide production

The production rate of carbon dioxide is proportional to the growth rate of methanotrophic bacteria in the filter bed. The biological degradation reaction of methane is given by Eq. (5).

$$CH_4 + O_2 \rightarrow xCO_2 + yH_2O + z\text{bacterial biomass} \quad (5)$$

In the case of total methane oxidation, supposing that no biomass is generated (theoretical reaction, with $x = 1$, $y = 2$ and $z = 0$), the mass ratio $P_{CO_2}/EC = 2.75$. Fig. 4 presents the rate of production of CO_2 (P_{CO_2}), expressed in g/m^3/h, as a function of the EC, also expressed in g/m^3/h, for the theoretical reaction case and for both the organic and the inorganic materials cases. For the organic bed case, the experimental data obtained all lie along the line represented by the relation; $P_{CO_2} = 1.6 \times EC + 17.1$, resulting in a line of slope 1.6. In the inorganic bed case, the experimental data lie along the line described by $P_{CO_2} = 2.5 \times EC + 2.2$, thus providing a line slope of 2.5.

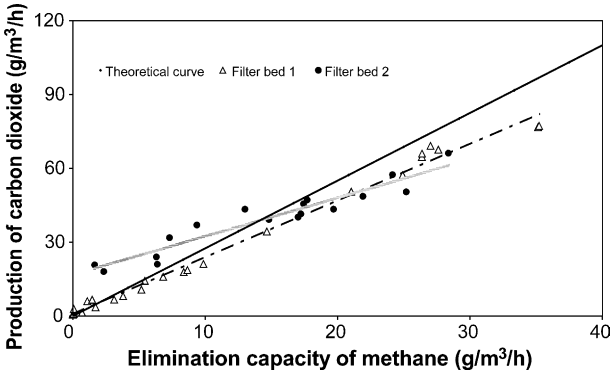

Fig. 4. Production of CO_2 as a function of elimination capacity for CH_4: one theoretical curve and two experimental curves: filter bed 1 (inorganic-based bed) and filter bed 2 (mature compost-based bed).

The greater the mass of CO_2 produced per gram of methane eliminated, the lesser is the proliferation of micro-organisms within the filter bed, thereby limiting the rate of bed clogging, in accord with Eq. (5). Throughout the range of inlet loads tested, as displayed in Fig. 4, filter bed 2 (the mature compost bed) produces less CO_2 related to methane elimination, in comparison to filter bed 1 (the inorganic material) ($1.6 < 2.5 < 2.75$). Consequently, filter bed 1 sustains lower levels of bed clogging because of the reduced rate of biomass production within it. This was also confirmed by visual observations.

Moreover, for a theoretical filter bed composed only of methanotrophs, the curve $P_{CO_2} = f(\mathrm{EC})$ should pass through the origin, meaning that a lack of methane consumption causes the carbon dioxide production to cease. The initial P_{CO_2} is higher for the filter bed 2 (mature compost material) (17.1 g $CO_2/m^3/h$) than for filter bed 1 (inorganic material) (2.2 g $CO_2/m^3/h$). This could mean that bacteria, non-specific to methane elimination, have a more significant presence in the organic bed.

3.3. Temperature and pressure drop

All bio-oxidation reactions are exothermic [19]. Consequently, a temperature increase between the inlet and the outlet ports of the biofilter is observed.

Fig. 5 presents, for both biofilters, the bed highest and lowest temperatures and therefore the temperature difference, as a function of nitrogen concentration. In the inorganic material, the temperature difference remains nearly constant during the whole experimental period and is less than 1 °C over the three stages of this biofilter. However, in the biofilter packed with mature compost material, the temperature difference was significantly greater, up to 2.8 °C, when the nitrogen concentration was greater than 0.5 g N/L.

The temperature difference in the organic-based bed, especially evident at an input nitrogen concentration of 0.75 g/L, was a consequence of the non-uniform degradation of methane in the biofilter 2. This non-uniformity was also observed considering the elimination capacity values for each of the three filter stages (data not shown). Indeed, these values showed that one stage was active (the first) with the highest temperature, while for the other two, smaller methane biodegradation was actually being performed, i.e., temperature actually decreased from the first bed to the last.

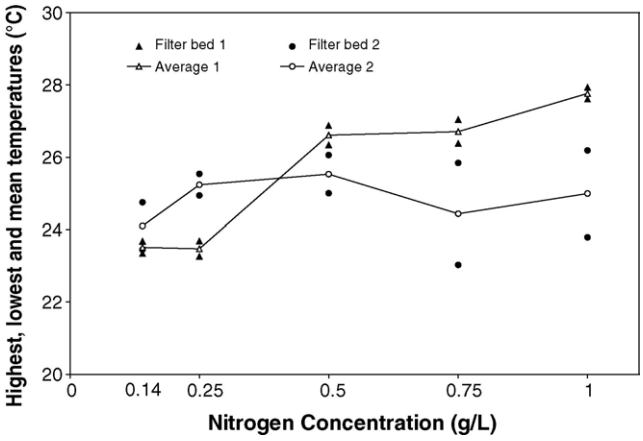

Fig. 5. Highest, lowest and mean temperatures in filter bed 1 (inorganic-based bed) and filter bed 2 (mature compost-based bed), as a function of nitrogen concentration.

However, the trend of these temperature differences remains similar to that of the elimination capacity, presented in Fig. 2 for both biofilters, the peak being observed at a nitrogen concentration of 0.75 g/L. This observation is proposed as proof that the filter bed temperature increase is a consequence of the biodegradation reaction, as reported by Delhoménie et al. [19].

Fig. 5 also presents the mean temperature of both biofilters as a function of input nitrogen concentration. Even if the temperature difference in the biofilter packed with inorganic material was lower than that with organic packing material, its mean temperature was higher for a nitrogen concentration equal to or exceeding 0.5 g N/L. For nitrogen concentrations at these levels, a difference in the performance of both biofilters has previously been noted (Figs. 2 and 3).

The pressure drops, measured during the starting period, were nil for both biofilters. For the mature compost-based filter bed, the pressure drop remained lower than 0.03 cm water per meter of biofilter height during the experiments. This value is low in comparison to others generally reported in the literature. During the planned period of biofilter operations, no significant pressure drop could be detected for the inorganic bed filter, again demonstrating that this filter packing material is a very suitable medium for use in methane elimination applications.

3.4. Dominant members of the microbial community in the inorganic-based bed biofilter

To find those microbial organisms most successful in colonizing the biofilm developing in the inorganic bed biofilter, the approach of DNA amplification by the polymerase chain reaction was utilized. The approach is semi-quantitative

and permits the taxonomic position of dominant members of a complex microbial community to be determined, without the necessity of bacteria growth in a medium. A rather large database exists for 16S rRNA sequences of procaryotes, allowing a rather precise identification of the various members of a microbial consortium.

Starting with the total DNA from a biofilm sample extracted from the inorganic bed biofilter, the dominant rRNA genes were amplified, using two primers corresponding to highly conserved portions of the 16S RNA sequence. Forty amplified DNA segments were taken for detailed analysis. Through the use of 10 different restriction enzymes, 12 different restriction profiles were identified, members of each profile were sequenced and the sequences analyzed with the BLAST program [17].

The most frequently (75%) recovered restriction profile corresponded, at the 98%-similarity level, to that of *Methylocystis parvus,* a type-II methanotrophic bacterium [18]. This microorganism appears to be the dominant methane oxidizer resident inside the biofilter. Other microorganisms were found in minor proportions, and included *Xanthomonas* sp. (5%), *Pseudoxanthomonas mexicana* (5%) and *Hyphomicrobium* sp. (2.5%). The latter is a methylotrophic microorganism, able to utilize methanol as the sole carbon source and is often found in mixed cultures with methane oxidizers [20].

4. Conclusion

The objective of this study was the measurement of the influence of nitrogen concentration, when supplied, as nitrate ions in a nutrient solution, during the biofiltration of methane. Two different filter beds were employed, one being composed of organic material (mature compost), the other of an inorganic material. The influence of the nitrogen nutrient input to the two filter beds, other parameters remaining the same, did not follow the same tendencies.

In terms of the elimination capacity parameter, the best results were obtained using the inorganic bed (29.2 g/m^3/h) for a nitrogen nutrient input concentration of 0.75 g/L, when the inlet load was between 65 and 70 g/m^3/h. In the mature compost-based filter bed, the maximum conversion performance ($\sim$19%) was achieved with a nitrogen concentration in the nutrient solution of 0.75 g/L.

The production of CO_2 was also measured for both bed types. These data lead us to conclude that, during the experimental trials, higher rates of CO_2 production per gram of methane eliminated, were observed occurring in the inorganic filter bed, thereby indicating that lesser amounts of biomass were being generated within this packing as compared to biomass production in the mature, compost-based bed, during the entire experimental period.

Microbial tests were separately conducted in the inorganic bed. These demonstrated that the dominant bacteria, present in the biofilter with that particular packing material, were *M. parvus*. Other measurements have shown that, in the same inorganic-based biofilter, the overall pressure drop is lower and bed temperatures are more uniform and at higher levels than those found in mature compost packed biofilters.

Acknowledgements

The authors are indebted to the Natural Science and Engineering Research Council of Canada (NSERC and NRCan First Greenhouse Gas Mitigation Technology Program) and to GSI Environnement Inc. (EMS Group) for their contribution to the project's financial needs. The authors would like to also express their gratitude to Dr. G. Soreanu for her comments and to Dr. P. Lanigan for text review.

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