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Publication date

2021

Document Version

Accepted author manuscript

Published in

Proceedings Sardinia 2021. 18th International Waste Management and Sustainable Landfilling

Citation (APA)

Buisma-Yi, S. C., Heijbroek, A. C., & Gebert, J. (2021). The effects of biochar on physical properties and methane oxidation capacity of cover soils from two Dutch landfills. In *Proceedings Sardinia 2021. 18th International Waste Management and Sustainable Landfilling : 11-15 October 2021* CISA Publisher.

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THE EFFECTS OF BIOCHAR ON PHYSICAL PROPERTIES AND METHANE OXIDATION CAPACITY OF TWO DUTCH LANDFILL COVER SOILS

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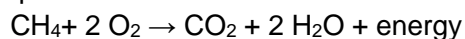
ABSTRACT: Designing methane oxidation systems (MOS) requires an understanding of soil physical properties and their changes to create an optimal habitat for methanotrophs. A short-term study was carried out to investigate the suitability of biochar as an additive to improve soil properties for use in MOS. The results from our batch experiments showed the effects of biochar on the particle size distribution, compaction and methane oxidation rates on two Dutch landfill cover soils collected from Braambergen landfill (fine-grained soil) and Wieringermeer landfill (more coarsely grained soil). When soils were amended with 6% (w/w) biochar, the particle size distribution curves shifted to enlarge the median diameter (D_{50}) in both soils. The methane oxidation rate increase was observed only on fine-grained soil with biochar with compaction, but both soils showed reduction in bulk density. Biochar addition enhanced methane oxidation rate in fine-grained soils when moisture content was kept constant at 17.8%, corresponding to a capillary pressure of >1000 hPa. Contrastingly, in coarsely grained soil, cumulated methane oxidation was reduced by 88.5% with biochar addition, despite the median diameter increase by 6%. Possibly, too high capillary pressures inhibited methanotrophic activity. The Procter density (D_{Pr}) decreased in both soils, but the optimum water content increased in the fine soil and decreased when biochar was added to the coarse soil. As expected, the biochar improved D_{50} , and optimum water content and methane oxidation in fine-grained landfill cover soil.

Keywords: Methane oxidation, landfill cover soil, biochar, compaction, particle size distribution

1. INTRODUCTION

Landfills are a significant contributor of anthropogenic methane emissions in the world (IPCC, 2006). Methane is a potent greenhouse gas with a global warming potential 25 times higher than carbon dioxide. Despite regulatory efforts to reduce landfill gas emissions, global methane emissions are expected to rise from increasing waste production and from countries relying on biomass landfilling as their primary waste management strategy. The most effective option to mitigate landfill methane emissions is to install active gas extraction systems and flaring or utilizing energy potential of the landfill gas, which is mandatory in many countries. However, when landfills reach low methane production potential, the residual gas can be mitigated by implementing biologically active methane oxidation systems (MOS) (Huber-Humer et al., 2008; Scheutz et al., 2009) in which methanotrophic bacteria oxidise the landfill methane to carbon

dioxide according to the following equation:



In these MOS, such as biocovers, biowindows or biofilters, different substrates (e.g., mineral soils, compost, wood chips, biochar etc.) are applied as material in the so called methane oxidation layer, either directly or in mixtures.

Biochar is a porous carbon-rich product made from pyrolysis of biomass produced in air-free conditions at 350-750 °C. Literature suggests that amending soils with biochar will have positive effects on soil health by changing physicochemical and biological properties of soils (Lehmann & Joseph, 2009). Biochar has the ability to reverse climate change and stimulate methane oxidation by increasing ammonia-oxidizing archaea and bacteria (Zhang et al., 2019). Further, biochars have large surface areas, increase soil porosity, water retention, reduce soil bulk density, and change soil texture (Yi et al., 2020). These characteristics make biochar a suitable substrate for MOS.

The performance of MOS relies greatly on the properties of the material used in the methane oxidation layer, as it governs both the geochemical conditions for methanotrophic activity (e.g., pH, salt content, and nutrient supply) and the soil's water and gas transport properties. The latter affects MOS performance in two ways: by impacting 1) the extent of desired spatial distribution of gas in an MOS, and 2) the rate of diffusive oxygen supply to the methanotrophs. Gas transport properties are strongly influenced by soil compaction; therefore, understanding how compaction modifies the physical properties of the material used in MOS is critical for MOS performance. Many studies reported biochar behaviour on agricultural soils and their benefits, where compaction is not required and discouraged.

Biochars have been used as a soil amendment to enhance CH₄ oxidation in landfill covers, where CH₄ removal rates were enhanced with biochar when incubated over 500 days (Yargicoglu & Reddy, 2017a). Biochar application rate of 2% (w/dw) at 5-6% gravimetric moisture content was correlated with enhanced methane oxidation rates (Yargicoglu & Reddy, 2017a), and the highest methane oxidation rates were observed on landfill cover soil amended with 10% (w/dw) biochar (Yargicoglu & Reddy, 2018). The results from these experiments showed that volumetric water content increased due to water production as a result of methane oxidation, and biochar surface pores enabled higher water sorption than the control that enhanced the population of methanotrophs (Yargicoglu & Reddy, 2017a, 2017b, 2018).

In this study, we investigated the suitability of biochar as an additive to improve the methane oxidation capacity of landfill cover soils in the Netherlands. Experiments were conducted to determine the effects of biochar on particle size distribution and compaction behaviour. In a pre-incubation experiment, the soils with and without biochar addition were exposed to methane to build up the methanotrophic community and methane oxidation capacity of the soil. Two Dutch landfill cover soils collected from Braambergen landfill (fine-grained soil) and Wieringermeer landfill (more coarsely grained soil) were amended with 6% (w/w) biochar produced from fir tree feedstock. It was hypothesized that, at a given degree of compaction, the addition of biochar would enhance the CH₄ oxidation capacity in both cover soils by increasing its air-filled porosity.

2. Materials and methods

2.1 Landfill cover soils and biochar

Landfill cover soils were collected from Wieringermeer landfill (Middenmeer, Netherlands) and Braambergen landfill (Almere, Netherlands) operated by Afvalzorg Deponie B.V. Selected properties of these soils are shown in Table 1. Braambergen soil (BRA) is a finer soil (silt loam) that contains significantly more clay and silt, and consequently less sand than Wieringermeer soil (WIE) with a coarse texture (loamy sand). BRA soil also featured a higher organic matter content, a higher optimum water content but a lower Proctor density. Both soils had been previously exposed to methane. WIE soil had been in place on a biocover test field (Geck et al., 2016) while BRA soil was used as cover soil in direct contact with the waste body, i.e. without an intercepting surface liner.

The biochar (Pyropower, Delft, Netherlands) was produced from disposed Christmas (fir) trees using a slow pyrolysis system at 500°C.

Particle size distribution of soils and soil-biochar mixtures was analysed employing a sieve analysis (ASTM, 2003). The standard Proctor test was used to find the relationship between soil water content and dry bulk density (ASTM, 2015).

Table 1. Characteristics of Wieringermeer (van Verseveld & Gebert, 2020) and Braambergen soil (Holland, 2020).

*TOC estimated from loss on ignition (4.47%).

Soil property	Wieringermeer (WIE)	Braambergen (BRA)
Clay (< 0.002 mm)	8.8%	21%
Silt (0.002 mm – 0.05 mm)	7.2%	52%
Sand (0.05 mm – 2.00 mm)	82.4%	27%
Gravel (> 2.00 mm)	1.6%	0%
Organic matter	TOC % = 1.3	TOC % = 2.3*
Proctor density	1.76 g/cm ³	1.70
Optimum water content (W_{opt} at D_{pr})	13.1%	17%
Soil texture (USDA soil taxonomy)	Loamy sand	Silt loam

2.2 Methane oxidation capacity

The cover soils were thoroughly mixed with 6% (w/w) biochar to provide a homogenous mixture. These samples are referred to as 6%BC+WIE (6% w/dw biochar added to Wieringermeer soil) and 6%BC+BRA (6% w/dw biochar mixed with Braambergen soil) herein. Control soils without biochar addition are referred to as WIE (Wieringermeer soil) and BRA (Braambergen soil).

Four 26 L barrels were prepared, each filled with ~5 kg of WIE, 6%BC+WIE, BRA and 6%BC+BRA soil. For the purpose of pre-incubating the soils for a later experiment, the soils were poured into the barrels loosely and not compacted in any way. The (gravimetric) moisture contents were adjusted to 5.9% (w/dw) for both WIE and 6%BC+WIE, and to 17.8% (w/dw) for BRA and 6%BC+BRA. These moisture contents represent estimated volumetric water contents corresponding to capillary pressures of 1000 hPa at a degree of compaction of 85%. The capillary pressures were estimated from the water retention curve using the van Genuchten model (van Genuchten, 1980) where the average hydraulic parameter values, the residual water content (θ_r) and the saturated water content (θ_s) were estimated from equations (1) and (2) using the quantity of clay, total organic carbon (TOC) and dry bulk density (ρ_{dw}) of the (van Genuchten, 1980; Vereecken et al., 1989).

$$\theta_r = 0.015 + 0.005 \text{ clay} + 0.014 \text{ TOC} \quad (1)$$

$$\theta_s = 0.81 - 0.283 \rho_{dw} + 0.001 \text{ clay} \quad (2)$$

The buckets were then sealed tightly and 2.6 L of pure CH₄ (99%) was injected into each bucket to produce a concentration of 9-11% CH₄ in air. The barrels were incubated at 19-20°C and CH₄ concentration was measured daily using Agilent Technologies 490 Micro-GC (Santa Clara, California, USA). When either CH₄ or O₂ were depleted, the barrels were opened ajar until the samples returned to ambient conditions (~18-24 h) and the cycle was repeated after weighing the buckets to ensure that the moisture contents remained the same and no evaporation occurred. The buckets were resealed, tested for ambient gas conditions using the GC before readjusting the barrel headspace to ~10% CH₄. Each repeated injection of CH₄ is referred to as a phase. A total of four phases are presented for each sample in this study.

Using the % (v/v) concentration of CH₄, the methane oxidation efficiency was calculated using:

$$CH_4 \text{ oxidation rate} = \frac{|\frac{\Delta C}{\Delta t}| VM_{CH_4}}{V_m M} \quad (3)$$

And the cumulative methane oxidation was calculated using:

$$\text{Cumulative oxidation } CH_4 \text{ by mass} = \sum_{i=0}^{j=i+1} \frac{|\frac{\Delta C}{\Delta t}| VM_{CH_4}}{V_m M} \quad (4)$$

Where $|\Delta C/\Delta t|$ is the absolute value of the slope between changes in methane concentration ΔC (%v/v) over changes in time (Δt), respectively. Oxidation rates were only calculated over the linear part of the slope with a Pearson's coefficient > 0.97 , hence representing zero order oxidation rates. V is the gas volume [L], M_{CH_4} is the molar mass of methane (16 g mol^{-1}), V_m is the molar volume (L mol^{-1}) assumed 24.4 L mol^{-1} at 293°K , and M is the dry mass of sample. The index i is phase and j is the total phase, where $j = 4$.

3. RESULTS AND DISCUSSION

3.1 Particle size distribution

Figure 1 shows the particle size distribution of pure biochar obtained from the manufacturer. The D_{50} of biochar was 7 mm, in the same diameter range as gravel ($>2 \text{ mm}$). About 60% of the bulk biochar had particle sizes larger than $>2 \text{ mm}$. Biochar is produced in a range of particle sizes ranging from a few nanometres to millimetres. The particle size of biochar depends on the particle size of the feedstock material and the pyrolysis temperature (Demirbas, 2004). Biochar produced from wood-based feedstock can provide the largest particle size fractions, where the average particle diameter (D_{50}) can range from 0.7 to 3.8 mm (Lim et al., 2016) or even larger as shown in this study. This is an important consideration since biochar particle sizes can control soil properties ranging from hydraulic improvement to microbial activity by changing soil pores.

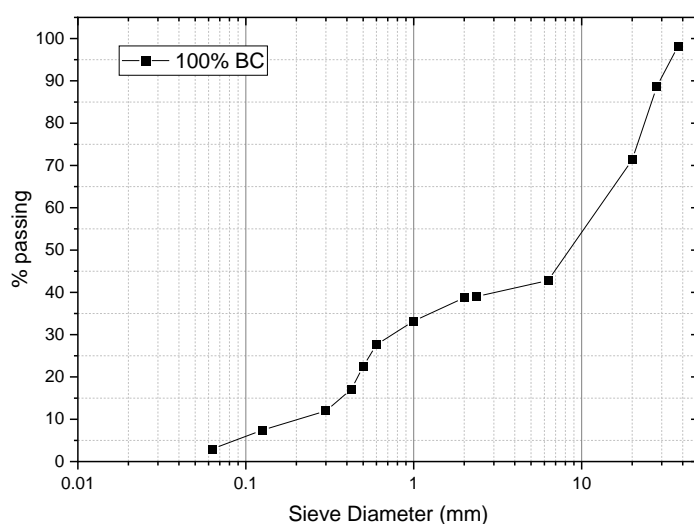


Figure 1. Particle size distribution of Pyropower biochar.

Biochar particle diameter at or below $\sim 0.5 \text{ mm}$ in sandy soil at 2% (w/dw) amendment increased water retention and pore size distribution in wood-based biochars (Yi et al., 2020). In this study, the applied biochar particle size was larger than 0.5 mm. It is unclear if larger biochar particles enhance or degrade pore size distribution of the bulk soil. This is dependent on biochar surface, particle size, and surface area. An important consideration is that biochar disintegrates, ages, breaks, which will influence microbial

activities (Lim et al., 2016). Biochar particle size and porosity is dynamic, which will further change soil properties.

After the biochar was added to the landfill soils at dry mass of 6% (w/dw), the particle size distribution curve shifted slightly to the right (Figure 2). The D_{50} increased in both WIE and BRA despite the soil texture differences. The D_{50} increased by ~6% when biochar was added in WIE (0.16 mm to 0.17 mm). In Braambergen, a larger shift (24%) in the mean diameter was observed. The D_{50} in BRA was 0.18 mm and adding 6% biochar to BRA, the D_{50} was 0.23 mm. Figure 2 shows that there are changes in the average particle diameter from biochar amendment. This may change the soil's gas transport and water retention properties as well as its inner surface area and therefore affect methanotrophic activity. It is expected that biochar addition to BRA soil, being more finely textured, will have a larger impact than in WIE soils since the difference in change for D_{50} is larger in BRA.

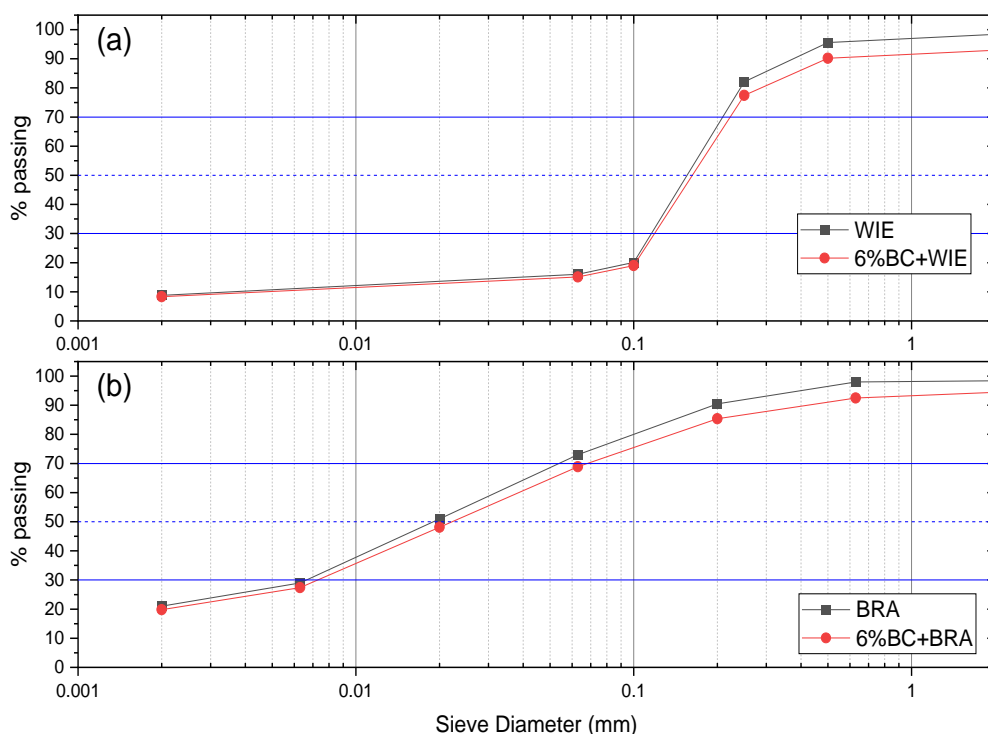


Figure 2. Particle size distribution curves of: (a) Wieringermeer soil (WIE) and 6% biochar in Wieringermeer (6% BC+WIE), and (b) Braambergen soil (BRA) and 6% biochar in Braambergen soil (6% BC+BRA). The dotted blue lines indicate the intersection of D_{50} , and the solid blue lines below and above the dotted lines indicate D_{30} and D_{70} intersections, respectively. Biochar mixtures do not sum up to 100% since >50% of biochar are > 10 mm.

When methane oxidation rates were evaluated in respect to soil particle sizes between coarse, medium and fine textures, the coarse grained soils had one order of magnitude higher CH_4 uptake rates than in finer soils (Dörr et al., 1993). Methane oxidation activity mostly occurs on soil diameter between 0.5 to 2 mm since larger diameters can enhance soil aeration, which is the dominant mechanism that drives CH_4 uptake. The methanotrophic bacteria, however, have a preference for attaching on mineral particles and methane oxidation activity can be found on smaller clay and silt particles since they can represent the bulk soil minerals (Bender & Conrad, 1994). As more research is performed to understand the effects of biochar particle diameter on soil properties in respect to methane oxidation, it is important to note that biochar particle size can be customized by pre- and post-treatment of biochar or the biomass if particle diameter of biochar becomes a critical consideration.

3.2 Compaction and proctor density

The results from the compaction test for WIE and 6%BC+WIE are presented in Figure 3a. In this soil, biochar addition decreased the Proctor density from 1.74 g cm^{-3} to 1.57 g cm^{-3} , and the optimum water content decreased slightly from 14% to 13.5%. Also for the fine-grained BRA soil, addition of biochar decreased the Proctor density from 1.69 g cm^{-3} to 1.45 g cm^{-3} , while in contrast to the coarsely textured WIE soil, the optimum water content increased significantly, from 17% to 24% (Figure 3b) requiring a higher optimum water content to achieve the same level of compaction.

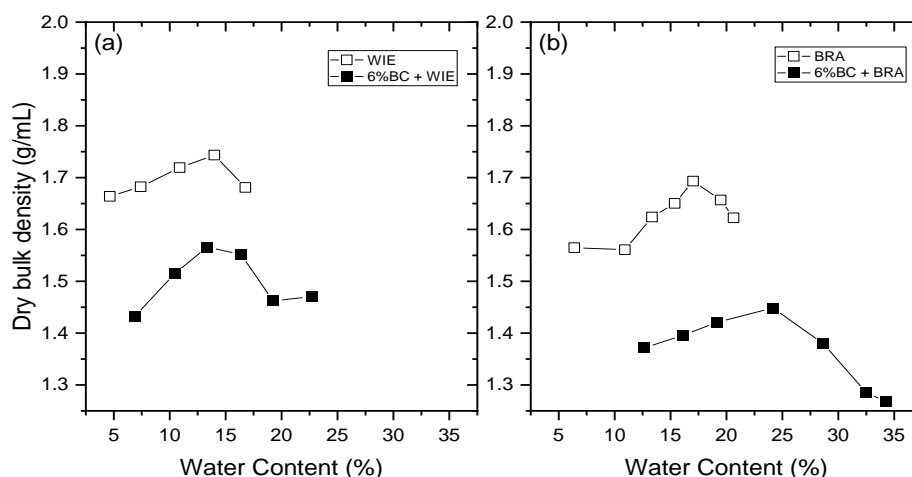


Figure 3. Standard Proctor compaction curves of: (a) Wieringermeer soil (WIE) and 6% (w/w) Pyropower biochar added to Wieringermeer soil (6%BC+WIE), (b) Braambergen soil (BRA) and 6% (w/w) Pyropower biochar added to Braambergen soil (6%BC+BRA).

Optimally, MOS material provides both a high porosity and water holding capacity. With biochar application, the effects of biochar application depends on the properties of biochar, the properties of the soil and how these two materials interact in the mixture, and therefore, the enhancement of methane oxidation will not always be met (Sethupathi et al., 2017). The compaction characteristics data show that the dry bulk density decreases when biochar is added to landfill cover soils (both in WIE and BRA), given the same compaction energy. This was expected since the specific particle density in biochar is lower than soils (Brewer et al., 2014). The reduction in soil bulk density demonstrates that biochar will alter soil particle packing, and potentially increase soil porosity.

Table 2. Dry bulk density of samples at 75%, 85%, and 95% compaction rates.

Relative compaction (C_R)	Wieringermeer (g cm^{-3})	6%Biochar + Wieringermeer (g cm^{-3})	Braambergen (g cm^{-3})	6%Biochar + Braambergen (g cm^{-3})
75%	1.31	1.18	1.27	1.09
85%	1.48	1.33	1.44	1.23
95%	1.65	1.49	1.61	1.38

The increase in soil water content is mostly observed when water is stored in both biochar pores and between the biochar-soil particles (Marsiello et al., 2015). The compaction procedure requires a brute force where a 2.5 kg hammer is dropped several times to pack into a standardised column. It was visually observed that biochar particles disintegrated while implementing the Proctor tests. The biochar breakage may change the particle size distribution and shift the D_{50} either higher or lower depending on the

amended soil texture. The relative compaction can also change as the compaction curves will not be reproducible due to the biochar particles breaking during the compaction tests (Lamprinakos & Manahiloh, 2019). Using the Proctor density, the dry bulk density of samples were calculated for three levels of compaction as shown in Table 2. The results from figure 3 are used to prepare samples at different compaction levels to test effective porosity and soil permeability. However, because biochars can break, it is unclear if the bulk density for 6%biochar/soil mixtures will exhibit the same desired compaction level.

3.1 Methane oxidation capacity

During the 35 d of incubation period, both WIE mixtures showed a similar trend in CH₄ oxidation rates in phase 1 (Figure 4a). However, no oxidation improvement was observed with biochar and the CH₄ oxidation rate decreased by 40% in phase 2 with WIE outperforming 6%BC+WIE by 123%. The oxidation rate declined per subsequent phase in WIE and 6%BC+WIE, with WIE having a 116% higher methane oxidation than 6%BC+WIE in phase 3. By phase 4, the difference between WIE and 6%BC+WIE was 20%. In the second and third phase, the CH₄ oxidation was significantly greater in WIE compared to biochar amended WIE (Figure 4a and Figure 5). In both soils, the CH₄ oxidation rates in phase 4 had decreased to below phase 1 values. The total oxidation rate was 252 mg CH₄-C kg⁻¹day⁻¹ for WIE and 97 mg CH₄-C kg⁻¹day⁻¹ for 6%BC+WIE (Figure 4c).

In contrast, biochar amendment to Braambergen soil enhanced methane oxidation overall (Figure 4b). In phase 1, the methane oxidation rate was 101% higher in 6%BC+BRA compared to BRA soil. This rate improvement difference decreased, but the subsequent oxidation rate difference was 73% (phase 2), 72% (phase 3) and 34% (phase 4), with 6%BC+BRA outperforming BRA soils. At ~35 d of incubation, the total oxidation rate of methane between the two samples were drastically different (BRA = 327 mg CH₄-C kg⁻¹day⁻¹ vs. 6%BC+BRA = 579 mg CH₄-C kg⁻¹day⁻¹) as shown in Figure 4c. Over the course of the experiment, methane oxidation rates continued to increase in BRA soil, with and without biochar. Possibly, the oxidation rate in the biochar-amended soil had reached its maximum after the third phase with the rate appearing to level off thereafter.

By comparison, maximum oxidation rates in the fine grained BRA soil exceeded those of the coarse grained WIE soil by up to factor 3 during the first four phases of the pre-incubation study. Loamy sand with a 25% (w/dw) moisture content can have a maximum oxidation rates of 2832 mg CH₄-C kg⁻¹day⁻¹ at an initial CH₄ concentration of 15% (v/v), and 4152 mg CH₄-C kg⁻¹day⁻¹ in 61% (w/dw) moisture content in silty loam at 5% (v/v) CH₄ concentration (Scheutz et al., 2009). The maximum oxidation rates in this study, during the 4 phases of incubation time, was more aligned with the oxidation rates of compost cover layer that was exposed with 8-10% (v/v) CH₄ concentration at 10-28% (w/dw) moisture content, where the maximum oxidation rate ranged between 60-384 mg CH₄-C kg⁻¹day⁻¹.

Figure 5 describes the ratio of CO₂ production and CH₄ consumption that elucidates the microbial community behaviour. The oxidation rates were decreasing in Figure 4a for both WIE mixtures, but the ratio between CO₂ and CH₄ fluctuated over the phases showing a V-shaped curve with or without biochar (Figure 5a). A ratio > 1 could imply that the methanotrophs are under stress and respire carbon storage and therefore release more CO₂ than the amount of CH₄ consumed. Alternatively, it could indicate that heterotrophic respiration dominates the carbon balance, rather than methane oxidation. However, in this study the ratio never rose above 1 (Figure 5a), and the rate of reaction is slower than BRA mixtures, possibly indicating longer incubation time to be necessary for microbial growth. At this current state, it is difficult to decipher the behaviour of biochar on WIE soil. It is possible that environmental factors may not be optimized that is delaying microbial activation.

In the BRA samples, there was a steady increase in methane oxidation rates. With biochar amendment, BRA soil started displaying an asymptotic curvature after phase 3 (Figure 4b). The steadily declining CO₂:CH₄ ratio (Figure 5) is an indication that there was continuous biomass growth (carbon assimilation), resulting in a lower release of CO₂ than would nominally correspond to the consumption of CH₄. In BRA soils, biochar augmented oxidation rates and led to a higher degree of carbon assimilation,

indicating that biochar enhanced methanotrophic biomass growth under these conditions.

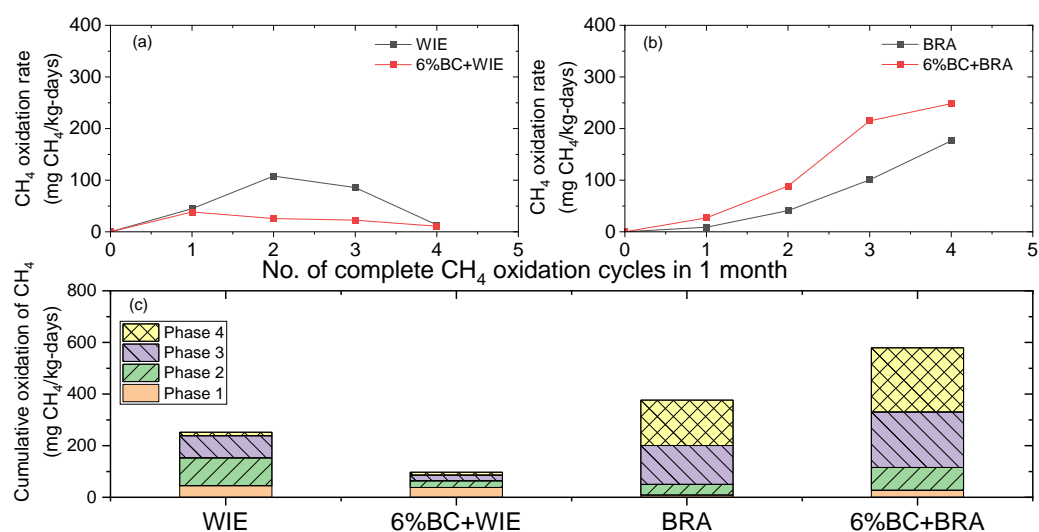


Figure 4. (a) Methane oxidation rates of Wieringermeer soil (WIE) and 6% (w/w) Pyropower biochar added to Wieringermeer soil (6%BC+WIE), (b) Methane oxidation rates of Braambergen soil (BRA) and 6% (w/w) Pyropower biochar added to Braambergen soil (6%BC+BRA), (c) Cumulative oxidation of CH₄.

Moisture content is one of the factors that affect CH₄ oxidation, where the optimum moisture content between 15.6-18.8% (w/w) can have the highest methane oxidizing capacity (Boeckx et al., 1996). The moisture contents were lower in WIE and 6%BC+WIE mixtures (~6%) than BRA and 6%BC+WIE mixtures (~18%). This appears as a large difference but reflects the water content at the same capillary pressure of 1000 hPa, averaged for bulk densities corresponding to 75%, 85% and 95% D_{Pr} of soil without biochar in light of the ensuing experiment involving different compaction levels. Since in the pre-incubation experiment soils were only poured into the barrels and not compacted, capillary pressures in the loose material were likely higher than if the soils would have been compacted, possibly leading to too dry (physiological) conditions in the sandy WIE soil. The condition may have been aggravated by addition of dry and coarse biochar. Water molecules and CH₄ may further have competed at the biochar pore level and may have had a negative effect on CH₄ adsorption in 6%BC+WIE (Sethupathi et al., 2017). Furthermore, the optimal moisture content was reduced by 0.5% with biochar in WIE soil after compaction (Figure 3), indicating that biochar in WIE soils may not necessarily enhance water holding potential despite biochar amended soils having lower bulk density than soils without biochar.

Even though soils were incubated in non-compacted condition under the same ambient condition, the moisture contents at ~6% for both WIE and 6%BC+WIE may be too low, or rather, capillary pressures may be too high, for enhancing oxidation rates compared to BRA mixtures. Furthermore, the D_{50} increased to 6% when biochar was added to WIE, whereas 24% increase was observed in BRA soil. WIE soils have large pore diameters, and to see an improved water storage, higher biochar application rates may be required. This was not observed in 6%BC+BRA, where the moisture content and D_{50} were much greater than the control, possibly stimulating environmental conditions for optimizing the methane oxidation rates. Further work requires gas transport and water retention properties to confirm the relationship between soil properties and methane oxidation rates with and without biochar.

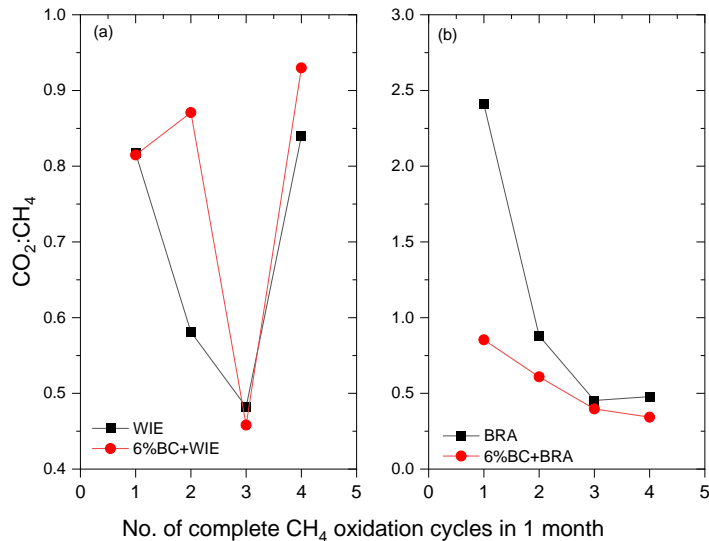


Figure 5. Ratio of % CO₂ produced to % CH₄ consumed in batch experiments of: (a) Wieringermeer soil (WIE) and 6% (w/w) Pyropower biochar added to Wieringmeer soil (6%BC+WIE), (b) Braambergen soil (BRA) and 6% (w/w) Pyropower biochar added to Braambergen soil (6%BC+BRA).

4. CONCLUSIONS

In this study, the effect of the addition of biochar on particle size distribution, compaction behaviour and CH₄ oxidation capacity was investigated in a coarse and in a fine grained landfill cover soil. So far, the following conclusions can be drawn:

- Particle size of biochar will change particle size distribution curves when added to soils. When D_{50} was enhanced by 24% with biochar in fine soil, the methane oxidation rate improved. However, D_{50} increase of ~6% in coarse soil did not improve methane oxidation during ~1 month incubation period.
- Compaction showed that biochar lowers dry bulk density, but optimal water content increase in the coarse soil was not observed. In a finer soil, the optimal water content increased and dry bulk density also decreased. The results show that less energy input is needed to achieve 85% compaction level for both cover soils.
- Methane oxidation rates do not always improve with biochar in sandy soils. It is suspected that methanotrophic activity in the sandy soil was moisture-limited, a condition which was not improved, but rather aggravated by biochar addition.
- Biochar was very effective in enhancing methane oxidation in finer soil, as evidenced both by higher methane oxidation rates and lower CO₂-CH₄ ratios, indicating higher methanotrophic population growth compared to the control soil.

In conclusion, soil texture seem to be a key factor governing methane removal capacity in this study. Upcoming research activities include the analysis of changes in water retention behaviour and effective gas diffusivity as well as an investigation into the CH₄ oxidation capacity at different levels of compaction, using the pre-incubated soils.

ACKNOWLEDGEMENTS

The authors would like to thank the TUDelft Bioengineering Institute for funding of this project. The authors would like to additionally thank the technical support staff at Department of Geosciences & Engineering at TU Delft: Roland Klasen, Michiel Slob and Wim Verwaal for their assistance, and Afvalzorg Deponie B.V. Afvalzorg Deponie B.V. for providing landfill cover soils used in this study.

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