Methanotrophic Methane Oxidation in New Biogeochemical Landfill Cover System

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Abstract: Municipal solid waste (MSW) landfills are regarded as one of the major sources of greenhouse gas (GHG) emissions across the world. In order to control these emissions, an innovative and sustainable biogeochemical cover system that consists of soil, biochar and basic oxygen furnace (BOF) slag is being developed to completely eliminate fugitive methane (CH_4) and carbon dioxide (CO_2) emissions from the landfills. The effectiveness of such cover systems is highly dependent on the survival and activity of methanotrophs under highly alkaline conditions induced by the presence of slag. In this study, a series of microcosm batch tests on landfill cover materials in different proportions were investigated to study the effect of cover materials on microbial CH_4 oxidation in the mixed as well isolated systems. Results demonstrated negligible CH_4 oxidation and substantial CO_2 sequestration when the BOF slag was integrated/mixed with soil (pH~7) and biochar-amended soil (pH~11). However, layered or separated cover material conditions (biochar-amended soil overlain by slag and soil overlain by slag) demonstrated promising CH_4 oxidation potential, thus concluding that extreme alkaline conditions inhibit the CH_4 oxidation. Overall, this study showed that a layered system consisting of the soil or biochar-amended soil layer overlain by BOF slag layer is optimal for CH_4 oxidation and subsequent CO_2 sequestration. Large column experiments and field test plots are being performed to evaluate the long-term performance of the proposed geochemical cover system under dynamic environmental (moisture and temperature) conditions.

Keywords: biogeochemical cover; BOF slag; biochar; methane oxidation; methanotrophs; carbon dioxide sequestration

INTRODUCTION

Landfills are the third largest source of anthropogenic CH_4 emissions in the United States. The landfill gas (LFG) typically comprises of 50% CH_4 and 50% CO_2, both of which are greenhouse gases impacting global climate change. Mitigation of CH_4 emissions has received greater attention for a long time, and many researchers evaluated reducing CH_4 emissions by studying the potential of CH_4 oxidizing bacteria present in the cover soil to convert CH_4 into CO_2 (Whalen et al. 1990; Kightley et al. 1995; Boeckx et al. 1996, Cao et al. 2011). In recent years, many researchers have focused on the use of biocovers that support microbial proliferation and enhance CH_4 oxidation. These biocovers typically employ organic-rich materials such as garden waste compost, yard waste, sewage sludge, peat, and biochar (Hummer and Lechner, 1999; Stern et al. 2007; Pedersen, 2011; Scheutz et al. 2011; Yargicoglu and Reddy, 2017) to enhance microbial CH_4 oxidation when placed alone or in amendments with soil.

In addition, an interest in utilizing industrial waste materials as components of landfill cover has also come into the limelight, due to their favorable physicochemical properties. Some of the industrial wastes that have shown potential landfill cover materials include paper mill sludge (Kovačić, 1996), coal fly ash (Nhan et al. 1996), bottom ash (Kim et al. 2016), and steel slag (Herrmann et al. 2010, Andreas et al. 2014) and they have been investigated as barrier or drainage layer depending on their hydraulic and geotechnical properties.

Steel slag, a byproduct from steel mills, has gained a significant attention in recent years, especially in the construction industry as an aggregate material and in environmental applications as media for contaminant adsorption and carbon dioxide sequestration. It is investigated in treating heavy metals from groundwater (Smith, 2003), phosphate removal from wastewater (Lu et al. 2008), heavy metals from acid mine drainage (Sheridan, 2014), and fertilizer/soil modifier in agriculture (Zhang et al. 2003; Kimio, 2015). Recently, Reddy et al. (2018) investigated use of BOF slag for sequestration of carbon dioxide from landfill gas emissions (Reddy et al. 2018).

An innovative, sustainable and practical biogeochemical cover system consisting of soil, biochar and BOF slag is being investigated to achieve zero emissions from the landfills (Reddy et al. 2018). The alkalinity and the presence of alkaline metals in BOF slag are conducive for CO_2 sequestration. However, it could be challenging for the microbial community in soil or biochar-amended soil to thrive under extreme alkaline condition induced by slag. The objective of this to investigate the effect of presence of these three landfill cover materials both on methanotrophic CH_4 oxidation and CO_2 sequestration.

BIOGEOCHEMICAL COVER CONCEPT

Biogeochemical cover is an innovative, low-cost landfill cover system consisting of steel slag in combination with soil and biochar (Reddy et al. 2018). Steel slag is a co-product of steel making process and basic oxygen furnace (BOF) slag is a type of steel slag, which is rich in alkaline minerals such as CaO, MgO, etc. The alkaline metal oxides present in the slag react with CO_2 forming stable carbonates. Many studies have explored the carbonation potential of steel slag for the mineral CO_2 sequestration. Past studies (Reddy et al. 2014; Yargicoglu and Reddy 2017) have shown promising potential in biochar-amended soil to mitigate CH_4 emissions by the enhanced methanotrophic oxidation of CH_4.

The biogeochemical cover aims to combine the carbonation potential of BOF slag with the methanotrophic CH_4 oxidation potential of biochar-amended soil to mitigate both the CH_4 and CO_2 emissions from the MSW landfills and ultimately leading to "Zero Emissions Landfill Cover". **Figure 1** shows the schematic of the steel slag and biochar amended-soil biogeochemical cover system. The proposed biogeochemical cover also has the potential to sequester hydrogen sulfide (H_2S) if present in the LFG as shown in Figure 1. The use of proposed biogeochemical cover in landfills will not only reduce the environmental concerns associated with the fugitive LFG emissions but also opens up a door for the sustainable management of steel slags which are generally stockpiled in the steel industry or landfilled.

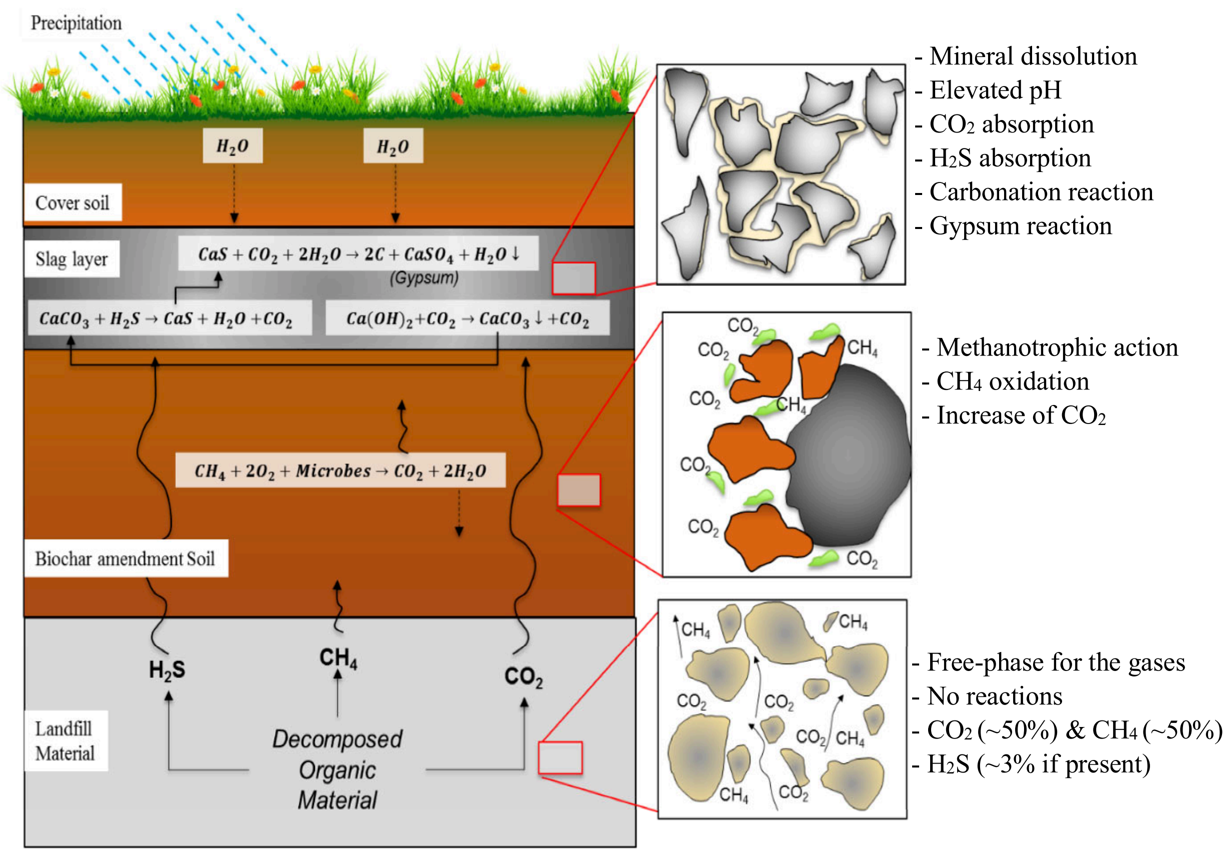

Figure 1: Schematic of biogeochemical cover system for zero emissions (Reddy et al. 2018)

Although the proposed biogeochemical cover offers wide range of environmental as well as economic benefits, it is of utmost importance to analyze various factors, which are crucial to the functioning of the coupled biogeochemical processes. A comprehensive laboratory testing program consisting of multiple tasks is undertaken for this purpose; this study presents the results from one of these tasks.

MATERIALS

Soil

Soil was collected from Zion landfill site, located in Greater Chicago area, Illinois, USA. Soil samples were collected from an interim cover layer at a depth of ~1 to 2 feet and were shipped to the Geotechnical and Geoenvironmental Engineering Laboratory at the University of Illinois at Chicago (UIC) where it was stored at room temperature (23 ±2°C). Soil samples were air dried (moisture content <0.5%), pulverized and screened through a 2 mm sieve prior to conducting the experiments.

Biochar (CE-WP2)

Biochar was obtained from a commercial vendor in Illinois, USA. The biochar used in this study was produced from pinewood pellets subjected to gasification at a high temperature of ~520°C. In this study, biochar in pellet form was used- fines were sieved and discarded. The biochar was oven-dried at 105°C to remove any moisture content before conducting the experiments.

BOF Slag

The BOF slag used in this experiment was obtained from Indiana Harbor East of Arcelor Mittal steel industry, located in Indiana, USA. All the tests were performed using the slag as obtained from the plant. The steel slag was also oven-dried at 105°C prior to conducting the experiments.

METHODOLOGY

Properties Testing

The specific gravity of the three landfill cover materials was determined in accordance with the ASTM D854. ASTM D422 was followed to determine the grain size distribution of each material, while Atterberg limits of soil were determined as per ASTM D4318. Hydraulic conductivity was determined according to the ASTM D2434 (for biochar and slag) and ASTM 5084 for soil using a flexible wall triaxial set up. The water holding capacity (WHC) of all the materials were determined using procedure as described in Yargicoglu et al. (2015). Each material under investigation (10 g) was soaked in 0.01 M $CaCl_2$ solution (L/S of 1:1) for 2 hours prior to measuring pH, ORP and electrical conductivity as per ASTM D4972. The pH meter was calibrated with standard buffers of pH 4, 7 and 10 prior to testing. The organic content of the materials was analyzed following the ASTM D2974.

Batch Incubation Tests

Mixed Systems

10 g of the total material (soil, biochar, and BOF slag) individually and in different proportions (**Table 2**) was placed in 125 ml-serum vials and adjusted to a moisture content of 20% (w/w) using deionized water. The vials were sealed airtight using butyl rubber septa followed by crimp cap. 20 ml of air from the headspace of each vial was replaced with equal volume of synthetic LFG

comprising of 50% (v/v) CH_4 and 50% (v/v) CO_2 to achieve a headspace concentration of ~5-6% (v/v) CH_4 and ~5-6% (v/v) CO_2 balanced in air (~88-90%). The change in the headspace concentration was determined by collecting and analyzing the gas samples on a regular basis using gas chromatography (GC) until the headspace concentration dropped to less than 1%. All the experiments were conducted in duplicate/triplicate along with the controls (gas with no material). The controls using only soil (sterilized for 2 hours using Napco Model 8000-DSE autoclave) were also tested to discern any microbial activity in the soil. The CH_4 oxidation rates were calculated from the linear regression analysis of CH_4 concentration versus elapsed time, based on the zero-order kinetics.

Slag Isolated Systems

Separate series of incubation experiments were conducted in which soil and biochar-amended soil were not mixed with slag, but slag existed separated in a cage. A steel cage of size 2" x 2" x 2" was used to contain the steel slag (10% of the total material) and placed inside the serum vial using nylon thread to isolate slag from the soil and biochar-amended soil. The material was adjusted to the desired moisture content of 20%. The vial was sealed airtight using butyl rubber septa followed by crimp cap. Similar procedure as mixed system was followed in the isolated system to achieve the headspace concentration of ~5-6% (v/v) CH_4 and ~5-6% (v/v) CO_2 balanced in air (~88-90%).

Gas Analysis

The gas samples were analyzed at regular time intervals and analyzed for CH_4, CO_2 and O_2 concentrations using an SRI 9300 GC equipped with a thermal conductivity detector (TCD) and CTR-1 column that separates N_2 and O_2 for simultaneous analysis of CO_2, CH_4, O_2 and N_2. Gas samples were withdrawn using 1 ml syringe where 0.5 ml of the sample was discarded and remaining 0.5 ml was injected into the GC to reduce any pressure effects due to sampling. A calibration curve for a minimum of three points was established using high purity standard gas mixtures ranging from 1% to 50% CH_4 and CO_2.

RESULTS AND DISCUSSION

The physical and chemical properties of BOF slag, soil and biochar used in this study are summarized in **Table 1**. The BOF slag tested consisted of ~74% sand-sized particles and was classified as SP (poorly graded sand) according to Unified Soil Classification System (USCS). The specific gravity of the BOF slag, soil and biochar were determined as 3.4, 2.57 and 0.6, respectively. The hydraulic conductivity of BOF slag and biochar were both approximately 10^{-3} - 10^{-4} cm/s, consistent with typical values for fine sands to loose silt (Holtz and Kovacs 1981). The soil was highly impermeable with a hydraulic conductivity in the order of 10^{-8} cm/s. The WHC of soil, BOF slag, and biochar were 43%, 20% and 52% (w/w), respectively. BOF slag was observed to be highly alkaline with pH 12.4. The ORP of all three materials were negative, demonstrating higher reducing capacity.

Both mixed and slag isolated systems were investigated for pH at the beginning of the experiments. Soil, biochar and BOF slag had pH of 7.6, 6.7 and 12.4, respectively (**Table 2**). Many studies on landfill cover soil have shown pH ranging from 4.3 to 9 (Gebert et al. 2009; Chi et al. 2015) with most of them at near neutral pH. The biochar is reported to have a wide range of pH, highly dependent on the type of feedstock used. Yargicoglu et al. (2015) showed pH ranging from 6.24 to 8.86 for five different types of biochar, produced from coconut charcoal, pinewood, aged oak, pinewood pellets, and 90% pine with 10% fir wood. The BOF slag was highly alkaline in nature due to the presence of basic oxides like CaO and MgO (Reddy et al. 2018; Bonenfant et al. 2008; Navarro et al. 2010). The amendment of BOF slag at 10% (total weight) in the mixed systems (slag-amended soil-biochar and slag-amended soil) decreased the pH by 1.4-1.5 units, keeping the overall pH at 10.9 and 11.03, respectively. This change in the pH was mainly due to high buffering capacity of the soil. However, the biochar-amended soil had the pH (7.4) close to the pH of the soil (7.6), indicating biochar had no major effect on the overall pH of the mixed materials.

Table 1: Physical and chemical characteristics of BOF slag, cover soil and biochar

Properties	**ASTM Method**	**BOF Slag**	**Soil**	**Biochar**
Specific Gravity	D854	3.5	2.57	0.6
Grain Size Distribution:	D422			
Gravel (%)		20.8	3.7	45
Sand (%)		74.2	14.7	54
Fines (%)		4.9	81.9	1
D_{50} (mm)			0.009	4.3
C_c		0.7	-	0.82
C_u		18	-	2.42
Atterberg Limits:	D4318			
Liquid Limit (%)		Non-Plastic	39	Non-Plastic
Plastic Limit (%)			22	
Plasticity Index (%)			17	
USCS Classification	D2487	SP-SM	CL	SP
Water Holding Capacity (w/w)		20	43	51.6
Dry Density (g/cm^3)		1.72	1.8	1.15
Hydraulic Conductivity (cm/s)	D2434	1.1×10^{-3}	5.4×10^{-8}	2×10^{-4}
Loss of Ignition (%)	D2974	1.6	5.8	96.71
pH (1:1)	D4972	12.4	7.6	6.5
Electrical Conductivity (mS/cm)	D4972	13.3	0.55	0.8
Redox Potential (mV)	D4972	-313.3	-53.8	-6.3

C_c=Coefficient of curvature; C_u=Coefficient of uniformity

Table 2: pH of the mixed and slag isolated systems

Substrates	pH
Soil (100%)	7.6
Soil (90%) + Biochar (10%)	7.4
Soil (90%) + Slag (10%)	11.03
Soil (80%) + Biochar (10%) + Slag (10%)	10.90
Soil (90%) & Slag in Basket (10%)	7.6 (Soil); 12.4 (Slag)
Soil (80%) + Biochar (10%) & Slag in Basket (10%)	7.4 (Soil + Biochar); 12.4 (Slag)
Biochar (90%) & Slag in Basket (10%)	6.7; 12.4 (Slag)
Slag in Basket (100%)	12.4

Figures 2(a) and **2(b)** show trends in CH_4 consumption, carbon dioxide production and oxygen consumption with time for soil and biochar-amended soil, respectively. An initial lag phase of 24-72 hours was observed, which could be mainly due to time needed for the adaptation of the microbial population to their environment. Thereafter, a gradual decrease in CH_4 concentration, increase in the CO_2 levels and decrease in the oxygen levels were observed, confirming CH_4 oxidation by the CH_4 oxidizing bacteria in both the systems. This was further confirmed using the controls (only sterilized soil and LFG) that showed no major change in the headspace gas concentration (not shown) confirming CH_4 oxidation by the naturally existing CH_4 oxidizing bacteria in the soil. The CH_4 oxidation rates calculated based on the zero-order kinetics were 89.2 µg/g/day and 79 µg/g/day, respectively, for soil and biochar-amended soil. The results from **Figure 2(b)** also suggests that the biochar had no major effect on the CH_4 oxidation process when amended with soil. Previous study from our research laboratory demonstrated promising results of biochar amendment in enhancing CH_4 oxidation rates in the long term, as the microbes take time for colonizing and acclimatizing (Yargicoglu and Reddy, 2017). The reason for negligible effect of biochar amendment in CH_4 oxidation in the current study could be attributed to the shorter duration of testing.

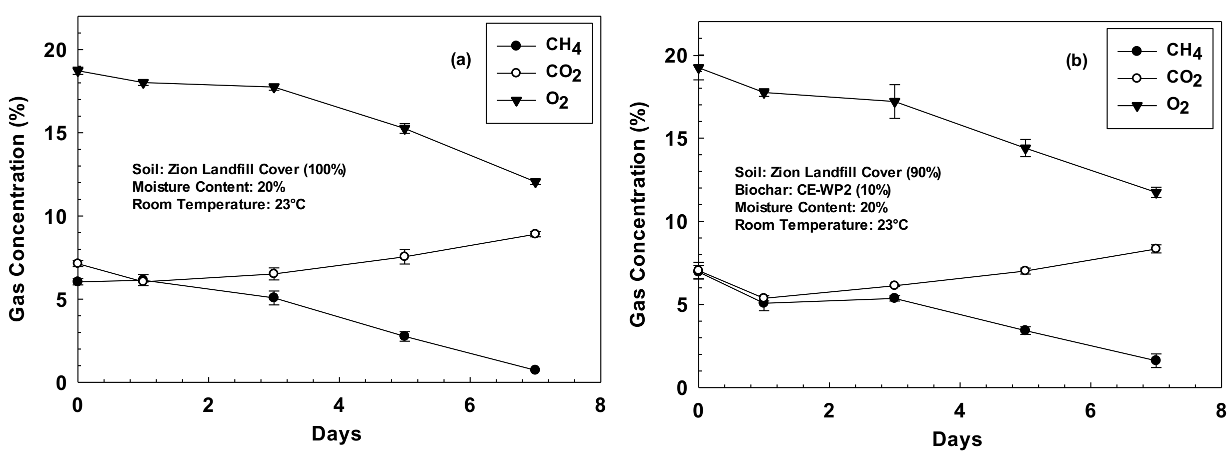

Figure 2: Removal of methane with: (a) Soil and (b) Biochar-amended soil

Figures 3(a) and **3(b)** show changes in the gas concentrations with time for slag-amended soil and slag isolated soil. The slag-amended soil showed complete removal of CO_2 within the first few hours of the experiment, but showed negligible change in the CH_4 concentration throughout the course of experiment suggesting inhibition of CH_4 oxidation activity of the CH_4 oxidizing bacteria, which could possibly be due to the high pH (11.03) of the system. However, in the slag isolated soil system, the slag was placed inside the steel mesh not in contact with the soil, wherein CH_4 oxidation by the soil and simultaneous CO_2 sequestration by the BOF slag was observed. The rate of CO_2 removal in the isolated system was slower when compared to the mixed system, which could likely be attributed to the diffusion limitations posed by the steel mesh. The CH_4 oxidation rates were 0.74 µg/g/day and 85.5 µg/g/day, respectively, for slag-amended soil and slag isolated soil.

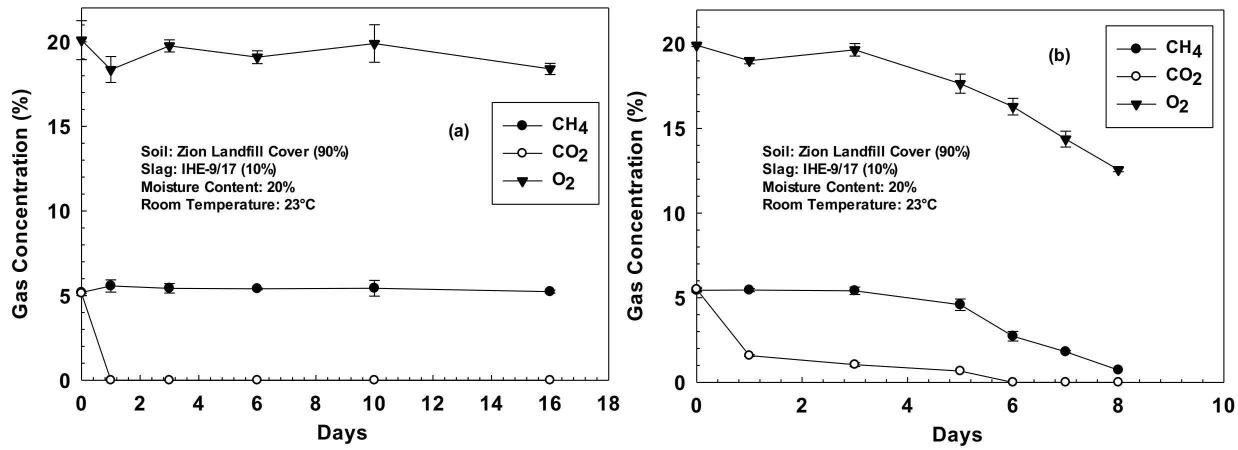

Figure 3: Removal of carbon dioxide and methane with: (a) Slag-amended soil (b) Slag isolated soil

Figures 4(a) and **4(b)** show changes in the gas concentrations with time for slag-amended soil-biochar and slag isolated biochar-amended soil. No change in the CH_4 concentration was observed in the slag-amended soil-biochar similar to the slag-amended soil, but complete removal of CO_2 in the presence of BOF slag was noted. However, in the slag isolated biochar-amended soil, change in CH_4 concentration with time was observed showing CH_4 oxidation along with a prolonged removal of CO_2. It is known that CH_4 oxidizing bacteria grow at a pH ranging from 5.5 to 8.5 in soils and sediments of different ecosystem (Dunfield 1993; Hutsch 1994; Scheutz and Kjeldsen 2004; Sherry et al. 2016; Han et al. 2016), although few methanotrophs growing in extreme environments such as soda lake and marine environments at pH 9-11 requiring NaCl for their growth (Kalyuzhnaya et al. 2008, Sorokin et al. 2000, Khmelenina et al. 1997) have been identified. Therefore, for an effective CH_4 oxidation to occur, slag isolated from the soil or overlain the soil is recommended so that methanotrophic activity is not inhibited by the high pH of the system. The CH_4 oxidation rate for these systems were calculated to be 0.98 µg/g/day (slag-amended soil-biochar) and 80 µg/g/day (slag isolated biochar-amended soil), respectively.

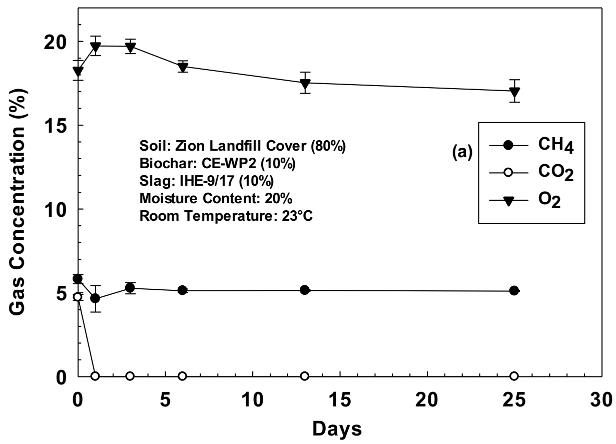

Figure 4: Removal of carbon dioxide and methane with: (a) Slag-amended soil-biochar (b) Slag isolated biochar-amended soil

Figure 5 shows trend in both CH_4 and CO_2 with time for BOF slag alone. The results show negligible removal of CH_4 and significant removal of CO_2 suggesting the BOF slag to be an invaluable material for CO_2 sequestration.

Overall, the three landfill cover materials studied demonstrated an effective CH_4 oxidation in the soil, slag isolated biochar-amended soil, slag isolated soil, and biochar-amended soil. It is important to note that negligible CH_4 oxidation in slag-amended soil-biochar and slag-amended soil was observed. Furthermore, BOF slag demonstrated significant potential in sequestering CO_2 in both mixed as well as slag isolated systems.

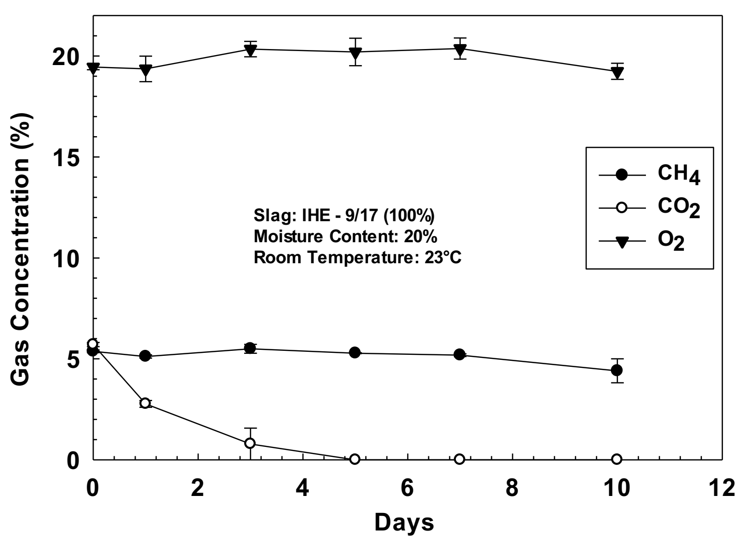

Figure 5: Removal of carbon dioxide using BOF Slag

CONCLUSIONS

Laboratory investigation on the landfill cover materials was conducted to study the effect of cover materials (soil, biochar-amended soil, and BOF slag) on the CH_4 oxidation and CO_2 sequestration. Our results demonstrated that the landfill cover soil was dominated by the CH_4 oxidizing bacteria and were responsible for the CH_4 oxidation. No negative impact on the CH_4 oxidation was observed when the soil was amended with biochar, but a negative effect was observed when the BOF slag was mixed with soil or biochar-amended soil. Nevertheless, BOF slag showed effective CO_2 sequestration in both mixed and slag isolated systems. Therefore, our results propose the use of slag-isolated soil or slag isolated biochar-amended soil systems for an effective CH_4 oxidation and simultaneous CO_2 sequestration in the biogeochemical landfill cover system. Column tests and field-scale evaluation of slag isolated biochar-amended soil cover systems are being performed in order to better understand the effect of BOF slag on microbial CH_4 oxidation under the long-term field environmental conditions.

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