CO_2 SEQUESTRATION USING BOF SLAG: APPLICATION IN LANDFILL COVER

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Abstract

Fugitive methane (CH_4) and carbon dioxide (CO_2) emissions at municipal solid waste (MSW) landfills constitute one of the major anthropogenic sources of greenhouse gas (GHG) emissions to the atmosphere. In recent years, biocovers involving the addition of organic-rich amendments to landfill cover soils is proposed to promote microbial oxidation of CH_4 to CO_2. However, most of the organic amendments used have limitations. Biochar, a solid byproduct obtained from gasification of biomass under anoxic or low oxygen conditions, has characteristics that are favorable for enhanced microbial oxidation in landfill covers. Recent investigations have shown the significant potential of biochar-amended cover soils in mitigating the CH_4 emissions from MSW landfills. Although the CH_4 emissions are mitigated, there is still considerable amount of CO_2 that is emitted to the atmosphere as a result of microbial oxidation of CH_4 in landfill covers as well as the CO_2 derived from MSW decomposition. Basic oxygen furnace (BOF) slag is a product of steel making has great potential for CO_2 sequestration due to its strong alkaline buffering and high carbonation capacity. In an ongoing project, funded by the U.S. National Science Foundation, the potential use of BOF slag in landfill covers along with biochar-amended soils to mitigate both CH_4 and CO_2 emissions is being investigated. This paper presents the initial results from this study and it includes detailed physical and chemical and leachability characteristics of BOF slag, and a series of batch tests conducted on BOF slag to determine its CH_4 and CO_2 uptake capacity. The effect of moisture content on the carbonation capacity of BOF slag was also evaluated by conducting batch tests at different moisture contents. In addition, small column experiments were conducted to evaluate the gas migration, transport parameters and the CO_2 sequestration potential of BOF slag under simulated landfill gas conditions. The result from the batch and column tests show a significant uptake of CO_2 by BOF slag for the tested conditions and demonstrates excellent potential for its use in a landfill cover system.

Keywords: CO_2 sequestration; BOF slag; biochar; carbonation; MSW landfills; landfill cover; landfill gas

1. INTRODUCTION

Global climate change and the rapidly increasing global population are currently some of the major concerns of the modern world. This has led to depletion of natural resources and increased generation of waste. In the U.S., landfills are the most dominant method of managing MSW. The MSW in landfills undergoes anaerobic decomposition to generate landfill gas that predominantly consists of methane (CH_4) and carbon dioxide (CO_2). The landfill gas (LFG) emissions are estimated to be one of the largest sources of greenhouse gas emissions into the atmosphere.

According to Resource Conservation and Recovery Act (RCRA) Subtitle D regulations, all new landfills are required to have active gas extraction systems to collect the LFG and prevent it from releasing into the atmosphere. In addition, a final cover with specific design requirements is made essential to minimize the infiltration into the waste as well as limit the escape of landfill gas into the atmosphere. However, the gas collection systems do not perform with 100 percent efficiency due to the limited radius of influence of each extraction well. Thus, there are always some fugitive emissions that cannot be targeted by the gas extraction systems. While serving the intended purpose, landfill covers also aid in reducing the fugitive emissions of LFG into the atmosphere to a certain extent by microbial oxidation of CH_4 to CO_2 in the presence of methanotrophs that naturally exist in the soil cover. But, the proliferation of these methanotrophs is limited and is not very effective in oxidizing large amounts of CH_4 emissions from the landfills.

In this regard, alternative covers called biocovers, involving the addition of organic amendments to the soil have gained great attention in the recent past. The addition of organic rich matter such as compost, digested sludge, biosolids, and peat moss enhances the microbial activity by providing the nutrients and microbial inoculums to the soil. But, the use of these materials has some limitations. For example, compost, dewatered sludge and biosolids like materials may themselves undergo anaerobic degradation adding to the already existing CH_4 and CO_2 emissions rather than reducing these emissions. Biochar is a promising material as an organic amendment in soil cover because of its unique characteristics (Reddy et al., 2014). Biochar is a solid byproduct derived from gasification or pyrolysis under low oxygen conditions. Biochar exhibits high internal porosity and high specific surface area which provides favorable conditions for microbial colonization and proliferation (Yargicoglu et al., 2015). In addition, it consists of stable organic carbon and doesn’t undergo any degradation. Recently, an extensive investigation involving lab-scale testing and field demonstration of the efficacy of biochar-amended soil cover system was performed (Sadasivam and Reddy, 2015; Yargicoglu and Reddy, 2017a, b, 2018). These studies confirmed the use of biochar as a potential organic amendment to mitigate CH_4 emissions from landfills quite successfully. However, one of the major limitations of the biochar amended soil cover system is that it does not address the fate of CO_2 which continues to be emitted into the atmosphere in undesirable amounts. In order to alleviate the problems from GHG emissions it is desirable to mitigate the CO_2 emissions as well.

The current study focuses on an innovative concept to mitigate both CH_4 and CO_2 emissions from MSW landfills by leveraging BOF slag. BOF slag is a product of the steel making process and it is known to possess unique characteristics suitable for CO_2 sequestration. Several different types of steel slag are obtained based on the steel type and steel making process (Shi, 2004). Currently, the coarser (larger aggregate sized) BOF slag material is utilized in asphalt paving due to its strong rutting resistance and durability, while the BOF slag fines are still largely stockpiled at steel plants. The BOF slag which has high alkaline buffering capacity can react with CO_2 in the presence of moisture to sequester CO_2 in the form of stable and insoluble carbonates. This process is generally known as the carbonation process.

In this study, a series of batch experiments were performed to estimate the carbonation capacity of a BOF slag. In addition, the effect of varying moisture contents on the carbonation capacity of the BOF slag was also studied. Furthermore, small column experiments were performed to evaluate the carbonation capacity of BOF slag under a continuous supply of simulated LFG emission conditions. The rate of carbonation and the breakthrough time are estimated based on the column experiments. The results show a substantial carbonation capacity of BOF slag and favor its use as a potential amendment in landfill cover system to mitigate CO_2 emissions.

2. MATERIALS AND METHODS

2.1 Steel Slag Characterization

Basic Oxygen Furnace (BOF) slag is produced when molten iron from blast furnace mixes with the fluxing agents (calcium oxide and dolomite) in the presence of 10-20% scrap steel melt and 99% pure oxygen blown at supersonic speed (National Slag Association 2013). According to U.S. Geological Survey, every year almost 15-20 million tons of ferrous slag and about 10-15 million tons of steel slag is produced. The BOF slag was collected from the Phoenix Services LLC facility at the Indiana Harbor East Steel Mill. BOF slag contains minerals that contain divalent cations of calcium, magnesium and iron that carbonate with time in the open environment. The mineral carbonation reaction of metal oxides (MO) can be simplified as:

$$MO + CO_2 \rightarrow MCO_3 + Heat$$

The mineral carbonation also results in the production of thermodynamically more stable products (carbonates). The natural mineral carbonation process could be accelerated and technologically viable in the presence of moisture and abundant availability of the minerals such as in BOF slag. The calcium silicates (and other silicates and aluminates) in the presence of moisture contributes to CO_2 sequestration (Huijgen et al., 2005) by the following chemical reactions.

$$CO_{2(g)} + H_2O_{(l)} \rightarrow 2H^+{}_{(aq)} + CO_3^{2-}{}_{(aq)}$$

$$CaSiO_{3(s)} + 2H^+{}_{(aq)} \rightarrow Ca^{2+}{}_{(aq)} + SiO_{2(s)} + H_2O_{(l)}$$

$$Ca^{2+}{}_{(aq)} + CO_3^{2-}{}_{(aq)} \rightarrow CaCO_{3(s)} \downarrow$$

In order to put BOF slag for beneficial use efficiently in a landfill cover system, it is necessary to understand its chemical, physical and geotechnical properties. Hence, a detailed characterization of the BOF slag was performed.

The elemental and mineralogical composition of the BOF slag was evaluated by XRF and quantitative XRD analysis (Grubb, 2017). In order to evaluate the potential toxicity of the use of BOF slag from leaching of toxic metals, both Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP) leaching tests were conducted as per the laboratory procedures established by United States Environmental Protection Agency (USEPA) under SW-846 (Grubb, 2017). **Table 1** provides the mineralogy of BOF slag used in this study. The TCLP and SPLP leaching test results of BOF slag are summarized in **Table 2**. Further, the basic chemical characteristics of the BOF slag were determined by measurement of pH, redox potential and electrical conductivity. The BOF slag used consisted majorly of oxides of calcium and iron. The leaching test results as shown in **Table 2** shows that the constituents leached from the material are within the regulatory limits.

The results indicated that the BOF slag was highly alkaline with pH in the range of 11.7-12.1. This is due to the presence of basic oxides like CaO, MgO and FeO in BOF slag. The specific gravity of the BOF slag was found to be 3.04 which is within the reported range of 3.0-3.46 in literature (Malasavage et al. (2012). The samples were analyzed for their particle size distribution (ASTM D422) and classified as poorly-graded sand. The hydraulic conductivity of the material was tested as per ASTM D2434 with a rigid wall permeameter. The hydraulic conductivity was evaluated to be in the order of 10^{-4} cm/s. The sample was also tested for its moisture retention capacity using the procedure described in Kinney et al. (2012). The physical, chemical and geotechnical properties of the BOF slag are shown in **Table 3**.

Table 1: Typical Mineral Composition of BOF Slag

Mineral	Mineral Formula	Percent Weight
Larnite	Ca_2SiO_4	20.6
Srebrodolskite	$Ca_2Fe_2O_5$	10.4
Iron Magnesium Oxide	FeO.76MgO.24O	6.7
Brownmillerite	$Ca_4Al_2Fe_2O_{10}$	5.8
Wuestite	FeO	5.4
Lime	CaO	4.1
Portlandite	$Ca(OH)_2$	6.5
Periclase	MgO	3.1
Magnetite	Fe_3O_4	3.0
Mayenite	$Ca_{12}Al_{14}O_{33}$	2.7
Quartz	SiO_2	0.5
Iron	Fe	0.3
Amorphous Material		31.1
Total		100.0

Table 2: Results from TCLP and SPLP Leaching Tests

Constituent	Symbol	RCRA Allowable Limit (mg/L)	TCLP Result (mg/L)	SPLP Result (mg/L)
Aluminum	Al		0.62	0.16
Antimony	Sb		<0.00031	<0.00016
Arsenic	As	5	0.00087	0.00029
Barium	Ba	100	0.14	0.12
Beryllium	Be		<0.00025	<0.00013
Boron	B		0.12	0.027
Cadmium	Cd	1	<0.00028	<0.00015
Calcium	Ca		2300	800
Chromium	Cr	5	0.011	0.002
Cobalt	Co		0.0034	0.0013
Copper	Cu		<0.005	<0.0025
Iron	Fe		0.031	0.011
Lead	Pb	5	<0.00041	<0.00020
Magnesium	Mg		0.077	<0.050
Manganese	Mn		0.005	0.00072
Mercury	Hg	0.2	<0.00005	<0.00005
Nickel	Ni		0.036	0.013
Potassium	K		0.76	0.66
Selenium	Se	1	0.0047	0.0019
Silver	Ag	5	<0.00025	<0.00013
Sodium	Na		6.4	4.8
Thallium	Tl		<0.00025	<0.00013
Vanadium	V		0.0058	0.00078
Zinc	Zn		0.035	0.024

Table 3: Physical, Chemical and Geotechnical Properties of Soil, BOF Slag and Biochar

Properties	Method	Soil	BOF Slag	Biochar
Physical				
Color	Visual	Brown, Brownish-grey, Grey	Grey	Black
Odor	Visual	Trace odor	Odorless	Odorless
Chemical				
Redox Potential (mV)	ASTM D4972-01	-37.7	-317.9	-6.3
Conductivity (μS/cm)	ASTM D4972-01	0.5	0.2	0.8
pH	ASTM D4972-01	7.04	11.7	6.5
Geotechnical				
Water Holding Capacity (w/w)	Kinney et.al. (2012)	45.93	40.54	51.55
Organic Content (%)	ASTM D2974-14	4.47	NA	96.71
Specific Gravity	ASTM D854-14	2.65	3.04	0.65
Atterberg Limits:	ASTM D4318-10		Non-Plastic	Non-Plastic
Liquid Limit (%)		35.0		
Plastic Limit (%)		20.34		
Plasticity Index (%)		14.66		
Grain Size Distribution:	ASTM D422-63			
Gravel (%)		12	0	45
Sand (%)		7	90.5	54
Fines (%)		81	9.5	1
Classification		CL	SP type	SP Type
Hydraulic Conductivity (cm/s)	ASTM D2434-68	2.75 x 10^{-8} @ 2.11 g/cc	4.2 x 10^{-4} @ 1.32 g/cc	2 x 10^{-4} @ 1.15 g/cc

2.2 Batch Tests

The BOF slag was evaluated for its CO_2 sequestration capacity by conducting a series of batch experiments. The batch tests were conducted to evaluate the effect of moisture conditions as moisture is an essential component in the carbonation process. In addition, these experiments were aimed at determining if there is any optimum moisture content for maximum amount of carbonation to occur in BOF slag. Since, the landfill covers predominantly remain under unsaturated conditions most of its lifetime, the water content range is selected such that lower limit is zero and the higher limit is the water holding capacity of the material. Also, all the tests were conducted under atmospheric temperature and pressure.

The BOF slag sample was dried at 100-110°C for 24 hours before it was used to conduct batch tests. The batch tests were conducted at 0%, 10%, 20%, 30% and 40% moisture content. To obtain the required water content, 1 g of the dried sample was mixed with 0g, 0.1g, 0.2g, 0.3g and 0.4g of distilled water separately and transferred to five separate 125 ml glass vials. Each vial was then purged completely with a synthetic landfill gas mix containing 50% CH_4 and 50% CO_2 by volume and closed with rubber septa and secured tightly with a metal crimp cap. For each of the water contents evaluated, the tests were conducted in triplicates. The samples were shaken vigorously before sampling the gas from their headspace. Gas samples were taken and analyzed using a gas chromatograph (SRI 9300 GC) equipped with a thermal conductivity detector (TCD) and CTR-1 column capable of separating N_2 and O_2 for simultaneous analysis of CO_2, CH_4, O_2 and N_2. Each 1ml of gas sample withdrawn from the vial was then reduced to 0.5ml sample volume before injection into the GC. This ensured the sample volume was within the acceptable limit for the GC and in equilibrium with the atmospheric pressure. A 3-point calibration curve was constructed for the GC using standard gas mixtures of 5%, 25%, and 50% methane.

2.3 Column Experiments

After the batch tests, long term column experiments were conducted to study the cumulative carbonation (CO_2 uptake) capacity of the BOF slag with continuous flow of the synthetic landfill gas (50% CH_4 and 50% CO_2) through the BOF slag packed in a small glass column with inlet and outlet ports. A schematic of the column experimental setup is shown in **Figure 2**.

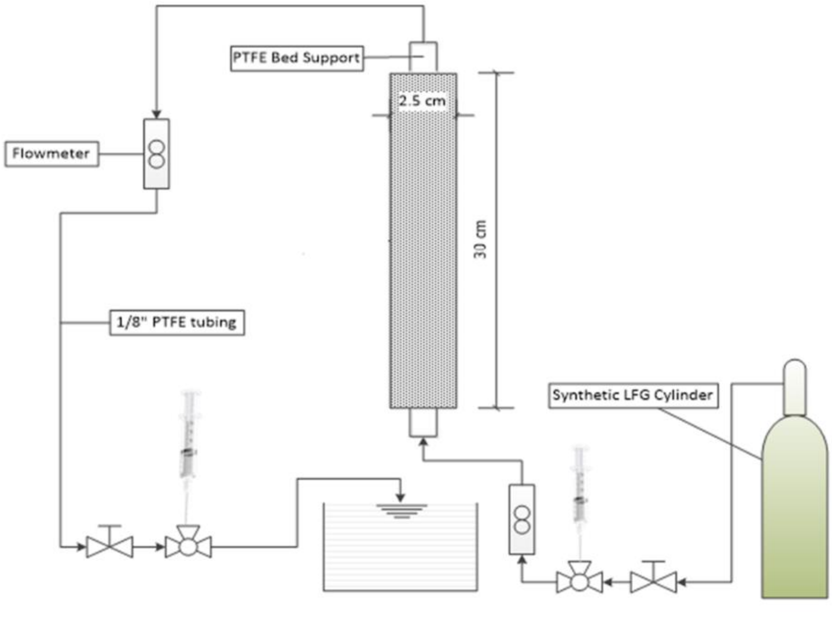


Figure 2: Schematic of Column Experimental Test Setup

An acrylic glass column of 30 cm height and 2.5 cm inner diameter was used. The column was filled with the BOF slag at 10% moisture up to its full length in two layers of 15 cm each with light tamping. It was secured with bed support mesh screen, end connections and screw caps at both ends. PTFE tubing was used to connect all components in the setup. Flow meters were installed at both ends of the column to control the influent gas flow rates and monitor the effluent flow rates. Gas samples were collected from both influent and effluent sampling ports at different time intervals until the breakthrough condition (where the inlet and outlet concentration of the gas becomes equal) was established.

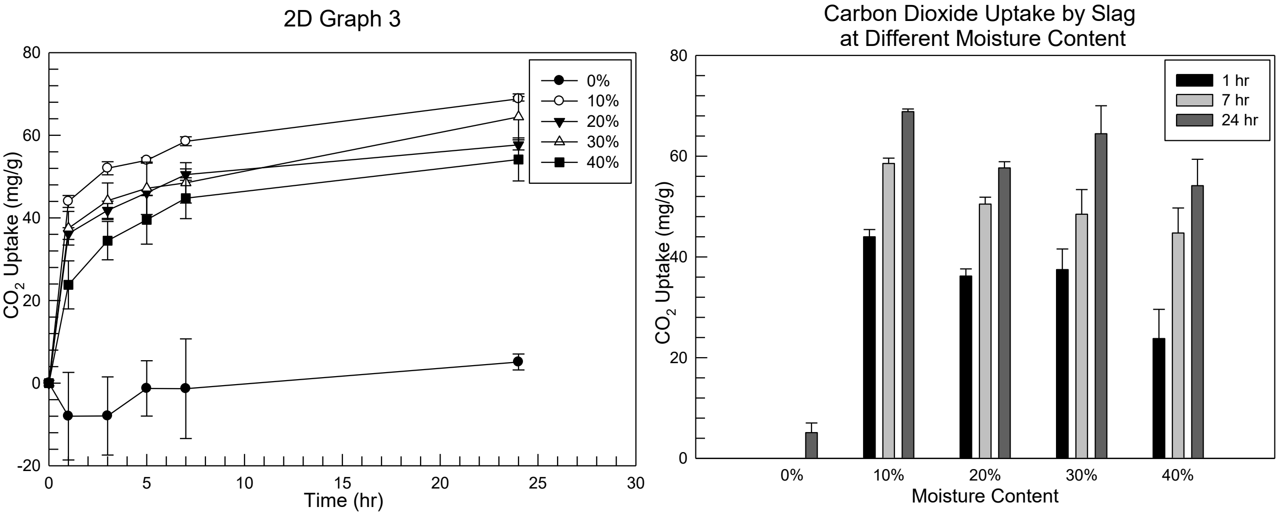
3. RESULTS AND DISCUSSION

3.1 Composition and Properties of BOF Slag

The chemical, physical and geotechnical properties of the BOF slag are favorable for its use in a landfill cover. The hydraulic conductivity of the BOF slag is in the order of 10^{-4} cm/s which is comparable with the hydraulic conductivity of biochar (10^{-4} cm/s). Due to the high hydraulic conductivity, the BOF slag and the biochar materials tested may not qualify as a barrier material. However, these materials could be used as individual thin layers in the cover soil or mixed with the cover soil for use in the landfill cover system. One of the characteristics that is unique to BOF slag is its high pH. The results from TCLP and SPLP confirmed that BOF slag is not a material of concern from the environmental risk standpoint as the results were found to be well within the regulatory limits (**Table 1**). Hence, the BOF slag is not hazardous and can be used as a cover material in landfills. The water holding capacity of BOF slag (40%) was comparable to that of the typical cover soil material (46%) indicating that the impacts of evaporative losses from landfill cover soils (such as desiccation cracking) could be minimum. BOF slag will not cause an additional odor since it does not contain any biodegradable material or sulfur and is characteristically odorless. Malasavage et al. (2012) conducted isotropically consolidated undrained triaxial shear tests on steel slag fines and reported a friction angle of 45.7° and cohesion of 48 kPa, while studying its geotechnical performance as synthetic fill materials. These high shear strength parameters of steel slag can as well enhance slope stability of a landfill cover. Additionally, the high specific gravity of BOF slag could also act as a factor that enhances the slope stability and reduces loss of material due to erosion.

3.2 Effect of Moisture Content on Carbonation of BOF Slag

Most of the previous studies that discuss the use of BOF slag for CO_2 sequestration focus on its industrial applications and involves carbonation of BOF slag under slurry conditions. This condition rarely exists in a landfill cover. The batch tests conducted at low moisture contents below saturation levels showed that substantial carbonation does takes place at moisture levels below saturation water content (**Figure 3**).



(a) **(b)**

Figure 3: Carbon Dioxide uptake by BOF Slag for Different Initial Moisture Contents

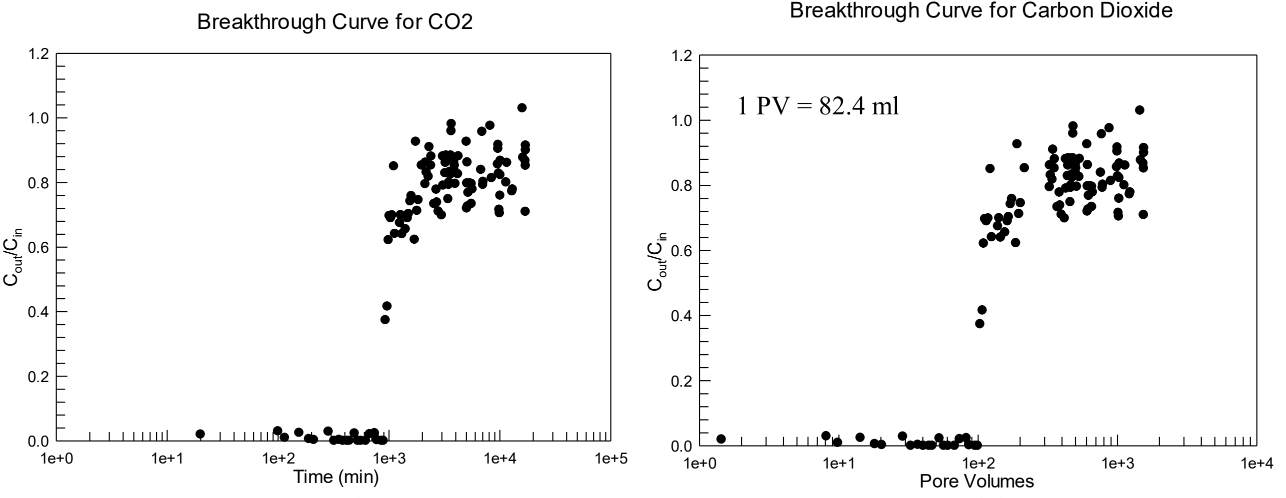
The results from the batch tests showed carbonation in the range of 53-68 mg of CO_2 per gram of BOF slag in 24 hours, with the maximum uptake of 68 mg/g. **Figure 3a** shows that the amount of CO_2 sequestered increased with time. However, the CO_2 uptake curve shows two distinct regions with different slopes indicating an initial rapid carbonation for a short period of time. The initial rapid carbonation could be due to the readily available free-lime and portlandite ($Ca(OH)_2$) in fine particles. The region of slow rate of carbonation for the rest of the time period could be from the lime diffusing out of larger particles. It could also be attributed to the limited access of CO_2 to the minerals due to the formation of carbonate precipitates over the mineral surface. A negligible amount of carbonation was observed in the absence of water as shown in **Figure 3b.** In addition, it was observed that there was no specific trend in the CO_2 sequestration with the moisture content and the CO_2 uptake was rather high and similar for all the moisture contents tested. This indicates that even moisture content as low as 10% could lead to substantial carbonation and is not significantly affected by the amount of moisture available in the system.

3.3 Gas Transport and CO_2 Uptake Capacity of BOF Slag

The breakthrough curve for CO_2 uptake with respect to time and pore volume of simulated LFG is shown in **Figure 4**. The breakthrough was obtained between 800-1,500 min and $C_{out} \geq C_{in}$ was achieved after 18,000 min. The cumulative uptake of CO_2 in the column test after 24 hours was in the same range as that of the 24 hour uptake in batch tests (see **Figure 5)**. The breakthrough curve was also plotted with respect to pore volume of gas for better understanding of CO_2 uptake in a large scale application. Accordingly, the breakthrough curve was obtained between 100-350 pore volumes (PV) of simulated LFG and the equilibrium was achieved at around 1,900 PV of gas. For the initial 100 PV the CO_2 was completely removed by the BOF slag. The breakthrough was achieved at around 100 PV after which the CO_2 uptake diminished rapidly, until a plateau was achieved at 350 PV where C_{out}/C_{in} was around 0.81. The curve maintained this ratio until there was no more uptake after 1,800 PV of CO_2 flow through the system.

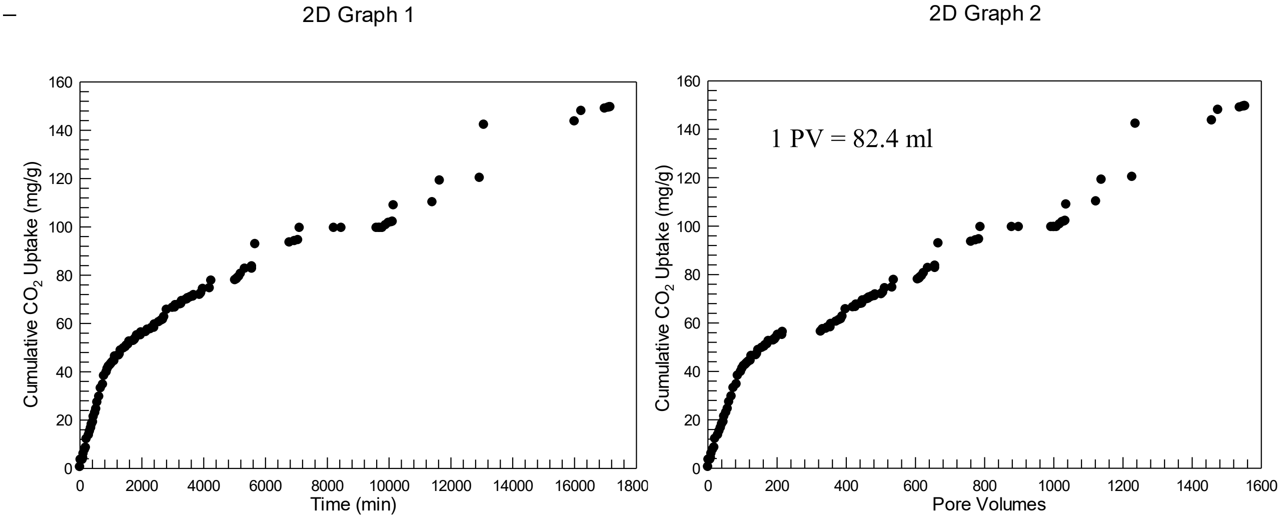
The initial reaction is attributed to the chemical reaction between the available free-lime and Portlandite in the material (which was ~4% and 6.5% respectively, see **Table 2**) with the CO_2 in the presence of moisture. After the exhaustion of the free lime, the reaction with other minerals such as calcium silicates (Ca_2SiO_4) could have been the source of continued uptake until the breakthrough. The adsorption and reaction with the interstitial minerals and other less reactive oxides and silicates that release calcium and/or portlandite could have led to the decent in the further reaction with CO_2 thus leading to the completion of the breakthrough curve. The lower reaction rates could also be attributed to the exhaustion of the limited moisture available in the system which was not replenished. Moisture plays a vital role in the carbonation reaction. It helps in dissolution of gas as well as interstitial CaO. Hence, the availability of persistent moisture on an actual landfill site could have higher capacity to capture more CO_2 during its lifetime. Also, the spreading of BOF slag over a larger area could generate more surface area allowing more interaction between gas and moisture leading to higher CO_2 sequestration.

In addition, a crust of carbonate precipitates was observed around the BOF slag material inside the glass column surface during the experiments which almost certainly limited mass transfer and reaction rates.



(a) **(b)**

Figure 4: Breakthrough Curve of Carbon Dioxide in Small Column Experiment with respect to (a) Time and (b) Pore Volumes



(a) (b)

Figure 5: Cumulative Uptake of Carbon Dioxide in Small Column Experiment with respect to (a) Time and (b) Pore Volumes

4. SUMMARY AND CONCLUSIONS

BOF slag has been extensively investigated for its potential use in different civil and environmental applications with the aim of preserving the natural resources and aim of mitigating the global CO_2 emissions (Motz and Geiseler, 2001) These studies have been useful in reducing the use of natural resources by replacing it with steel slag in various forms, alleviating the amount of steel slag stockpiling at the steel plants. There have been recent efforts at the potential use of steel slag in landfills, but as a drainage material in the landfill covers. This study explores the use of BOF slag to mitigate the CO_2 emissions from MSW landfills by leveraging the mineral carbonation process of BOF slag. In this regard, a series of batch tests were conducted to evaluate the amount of CO_2 that can be sequestered by the BOF slag. Furthermore, the carbonation and CO_2 sequestration under varying moisture contents was conducted to evaluate the effect of moisture content on the carbonation capacity of the BOF slag. Small column experiments simulating the flow of landfill gas through BOF slag were conducted with the optimum moisture content as derived from the batch test results.

Following conclusions could be drawn from this study.

- The BOF slag was found to have characteristics suitable for its use in a landfill cover based on the physical, chemical and technical characterization performed. The BOF slag used was classified as non-hazardous based on the TCLP and SPLP test results.
- The results from the batch tests showed that the carbonation capacity (CO2 uptake) of the BOF slag was about 68 mg/g within 24 hours. It was also observed that, for the range of moisture content tested, the CO2 uptake in 24 hours from the batch tests was comparable to the cumulative uptake of CO2 in column test.
- The cumulative uptake of CO2 from the column experiments is a conservative estimate since there was an exhaustion of the moisture available in the system for carbonation reaction over the course of the column test. The limited moisture availability in a continuous gas flow system could hinder the maximum possible carbonation of the BOF slag used in the column.
- Further studies are being performed to analyze the carbonation mechanism and evaluate the effects of various system parameters on carbonation capacity of BOF slag, including field landfill gas flow and environmental conditions.

Acknowledgements
This project is funded by the U.S. National Science Foundation (CMMI # 1724773), which is gratefully acknowledged.

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THESSALONIKI, JULY 2018

International Confernece Protection and Restoration of the Environment XIV
Proceedings (MEMORY STICK)

ISBN: 978-960-99922-4-4