around 50% of the landfill gas (LFG). Recently, Reddy et al. (2018a) proposed the concept of innovative biogeochemical soil cover, which can mitigate both CO_2 and CH_4 emissions from MSW landfills.



Figure 1: Schematic of batch experiment

Many studies have reported the CO₂ sequestration potential of alkaline industrial residues. Slags from steel making are alkaline, rich in calcium, and have demonstrated significant CO₂ sequestration potential (Huijgen et al. 2005; Bonenfant et al. 2008). Among the various types of steel making slags (basic oxygen furnace (BOF), electric arc furnace (EAF), ladle furnace (LF)), BOF slags have shown promising CO₂ sequestration potential (Chang et al. 2011). Chang et al. (2011) also evaluated the carbonation potential of steel slag under different operational conditions such as reaction time, temperature, and CO₂ flow rate. Liquid to Solids ratio (L/S ratio) also has been an important parameter affecting carbonation of steel slag. Different studies have reported different values of optimum L/S ratios for carbonation of steel slags based on their experimental conditions (Huijgen et al. 2005; Baciocchi et al. 2009; Sarperi et al. 2014, and Reddy et al. 2018b). However, many of these studies simulated industrial processes which are quite different than landfill conditions.

In this study, the authors investigated the effects of moisture (expressed by L/S ratios) on the CO_2 sequestration capacity of BOF slag exposed to simulated landfill gas conditions (50% CO_2 and 50% CH_4). The main aim of the study was to examine the performance of the BOF slag in CO_2 sequestration under various moisture conditions that may prevail in the landfill cover due to seasonal variations.

MATERIALS AND METHODS

Material Characterization

The BOF slag used in this study was obtained from Indiana Harbor East Steel Mill (supplied by the Phoenix Services, LLC). The BOF slag was crushed and screened at the plant with a maximum size of 10 mm before being supplied to the laboratory for the study. The physical and geotechnical characterization of the BOF slag was carried out following ASTM standard procedures. The specific gravity of the BOF slag was determined as per the ASTM D854 standard procedures. The grain size distribution was obtained from sieve analysis following ASTM D422 standard procedures. The water holding capacity was measured following standard

procedures as per ASTM D2980. The loss on ignition value was obtained according to the ASTM D2974 standard procedures. The pH of the BOF slag was measured using an ORION Model 720A pH meter at a liquid to solid ratio of 1:1. The electrical conductivity of the BOF slag was measured using a Corning 311 Conductivity meter.

Table 1: Physical and geotechnical properties of BOF slag						
Properties	Method	BOF slag				
Specific Gravity	ASTM D854	3.46				
Grain Size Distribution:	ASTM D422					
Gravel (%)		20.8				
Sand (%)		74.2				
Fines (%)		5.0				
$D_{50}(mm)$		1.5				
Cc		0.7				
Cu		18				
USCS Classification	ASTM D2487	SP-SM				
Water Holding Capacity (w/w)	ASTM D2980	20				
Loss on Ignition (%)	ASTM D2974	1.6				
pH (at L/S of 1:1)	ASTM D4972	12.1				
Electrical Conductivity (mS/cm)	ASTM D4972	13.3				

The chemical/mineralogical characterization of the BOF slag was achieved by X-ray powder diffraction and Rietveld quantification analyses. The morphological characterization of the BOF slag before and after carbonation was carried out by Scanning Electron Microscopy (SEM) analysis. The SEM analysis was performed by a JEOL JSM-6320F High Resolution Scanning Microscope.

Batch Experiments

Batch experiments were performed on the as-received BOF slag samples. Four 1 g specimens of oven dried BOF slag were mixed with 0, 0.1, 0.15 and 0.2 g of deionized water in four 125 ml glass vials to obtain the L/S ratios of 0, 0.1, 0.15 and 0.2. The upper limit of the moisture content used in the experiment was chosen based on the water holding capacity of the slag which was 20% (w/w). The glass vials with BOF slag were purged with synthetic LFG mixture (50% CO₂: 50% CH₄ (v/v)) under normal atmospheric condition and room temperature. Immediately after purging, the vials were sealed with a rubber septa and secured with a metal crimp cap to prevent any leakage of the gas. The vials were rigorously stirred to enhance liquid/gas mass transfer. Fig. 1 shows the schematic diagram of the batch test set up. Gas samples from each vial were analyzed by gas chromatography (GC) using a SRI 9300 GC equipped with a thermal conductivity detector (TCD) and CTR-1 column capable of separating N₂ and O₂ for simultaneous analysis of CO₂, CH₄, O₂ and N₂. One ml of gas samples from each vial were extracted in a 1 ml syringe which was then reduced to 0.5 ml before analyzing the gas sample to eliminate the pressure effects on the gas concentrations detected by GC. The GC was calibrated with three standard CH₄-CO₂ gas mixture of 5%, 25% and 50% (v/v) concentrations. Considering that most of the carbonation reactions occurred in the first few hours, the tests were performed for 24 hours, and the gas samples were analyzed at intervals of 0, 1, 3, 5 and 24 hours. All the bath experiments were performed in triplicate.

RESULTS AND DISCUSSION

Material Characterization

The physical and geotechnical properties of the BOF slags are summarized in Table 1. The grain size distribution curve is shown in Fig. 2. The grain size distribution shows that the BOF slag was silty sand type material with mean particle size of 1.5 mm. The BOF slag has low fines content. The pH of the slag is high which shows alkaline nature of the BOF slag. The loss on ignition value is low which indicates that the BOF slag has not undergone much aging and hence, is in reactive form. The specific gravity of the slag is 3.46 which is relatively high and can be attributed to the higher iron content.



Table 2: Chemical composition (major oxides) of BOF slag								
Sample	CaO (wt	SiO ₂ (wt	Al_2O_3	Fe_2O_3	MgO	SO ₃ (wt	LOI	
	%)	%)	(wt %)	(wt %)	(wt %)	%)		
BOF Slag	29	10.55	3.8	22.25	9.3	0.2	3.65	

Table 2 shows the major oxides present in the BOF slag, and Table 3 shows the mineralogical composition of the BOF slag. The major constituents of the BOF slag are CaO (29%), SiO₂ (~11%), Al₂O₃ (~4%), Fe₂O₃ (~22%) and MgO (~9%). High alkalinity is attributed to the high CaO content of the slag which is associated with the production process of the BOF slag. The major mineral phases present in the BOF slag are free lime (CaO, 2.4%), portlandite (Ca(OH)₂, ~2.5%), larnite (Ca₂SiO₄, ~12%), calcium ferrite (Ca₂Fe₂O₃, ~14.5%) and Mg-wuestite ((Mg,Fe)O, ~5.3%). The maximum theoretical carbonation potential of the BOF slag is 228 mg CO₂/g BOF slag which was calculated based on the total CaO content of the slag (29 wt%) and molar balance of the following reaction:

$$CaO + CO_2 \rightarrow CaCO_3$$
 (1)

The free lime content is relatively low in the BOF slag. The CaO is generally bound with the mineral phases in the form of calcium silicates and aluminates.

Table 3: Mineralogical composition of BOF slag						
Mineral	Formula	BOF slag (wt				
		%)				
Lime	CaO	2.4				
Portlandite	Ca(OH) ₂	0-4.4				
Larnite	Ca ₂ SiO ₄	11.7-12.6				
Calcite	CaCO ₃	0.8-1.1				
Srebrodolskite	$Ca_2Fe_2O_5$	14.2-15				
Magnesioferrite	MgFe ₂ O ₄	6-6.5				
Iron Magnesium Oxide	Fe0.76Mg0.24O	5.2-5.4				
Wuestite	FeO	3.4-3.5				
Periclase	MgO	0.7-0.8				
Brucite	$Mg(OH)_2$	0.4				
Mayenite	$Ca_{12}Al_{14}O_{33}$	2-2.9				
Akermanite	Ca ₂ MgSi ₂ O ₇	2				
Merwinite	Ca ₃ Mg(SiO4) ₂	1.8-2.1				
Katoite	$Ca_3Al_2(OH)_{12}$	1.7-1.8				
Amorphous Material		43-43.7				

Note: Analyzed by PMET, Inc., PA

Batch Experiments

The effect of moisture content on the CO₂ sequestration potential of the BOF slag was evaluated by performing batch experiments at different L/S ratios under landfill gas conditions (50% CH₄: 50% CO₂ (v/v)). The gas (CO₂ and CH₄) removal capacity was calculated by the difference in the initial concentration (C_{in} at t=0) and the concentration at time t (C_t) of the gas in the vial (C_{in}-C_t). The concentration of the gas (vol %) as obtained after analyzing in GC was converted to the concentration in terms of mg/l applying ideal gas law (PV=nRT). Considering volume of the gas is equal to the volume of the vial (125 ml), and the initial mass of the BOF slag taken was 1 g, the concentration of the gas was obtained in terms of mg/g as shown in Eq. (2).

Gas concentration
$$\left(\frac{mg}{g}\right) = \frac{mg}{l} \times \frac{0.125l}{1g}$$
 (2)

Fig. 3 (a) shows CO_2 and CH_4 removed in 24 hours. Fig. 3(a) shows negligible CO_2 removal in dry state (L/S =0) followed by significant increase in the removal in the presence of moisture. CO_2 removal shows an increasing trend from L/S of 0 to 0.15. The CO_2 removal at L/S from 0.15 to 0.2 were comparable. The BOF slag also showed some amount of CH_4 removal however, the removal is significantly lower (<10 mg/g). This could be attributed to the adsorption of CH_4 on the slag surface due to the presence of alumina (Al_2O_3) (Li et al. 1994). The 24 hours CO_2 removal capacity of the BOF slag was in the range of 32-45 mg CO_2/g BOF slag with optimum removal at L/S in the range of 0.15 to 0.2 (g/g).



Figure 3. Effect of moisture content on the removal of CO₂ and CH₄ by BOF slag at different liquid/solid ratio (L/S) exposed to synthetic landfill gas mixture (a) cumulative removal in 24 hours (b) removal of gas at different points of time until 24 hours

Various researchers have shown different optimum L/S ratios for carbonation in their experimental conditions. Huijgen et al. (2005) showed L/S ratio of 2 (kg/kg) as the optimum moisture content for carbonation of Linz Donawitz steel slag (LD) slag in slurry phase in their reactor. At L/S ratio higher than 2 (kg/kg), they observed a reduction in degree of carbonation with increase in moisture content. Baciocchi et al. (2009) achieved the highest CO₂ removal at L/S ratio ranging from 0.3 to 0.4 l/kg for stainless steel slag exposed to 100% CO₂. Sarperi et al. (2014) achieved optimum CO₂ removal at L/S ratio in the range of 0.05 to 0.2 l/kg for BOF slag exposed to mixed gas of CO₂ and CH₄. Su et al. (2015) observed highest carbonation of BOF slag at L/S ratio of 2-10 (g/g) in their experimental conditions. These studies show variable optimum moisture content range for CO₂ removal.

Among the studies carried out by various researchers, the study by Sarperi et al. (2014) was closest to our study. The L/S ratio for maximum CO₂ removal reported by Sarperi et al. (2014) was 0.1 l/kg whereas our study showed maximum CO₂ removal at L/S ratio of 0.2 g/g. Study by Reddy et al. (2018) also showed maximum CO₂ removal at a moisture content of 10% (w/w) or L/S ratio of 0.1 g/g. The difference could be due to the difference in the mineralogy of the BOF slag under study. The BOF slag used by Reddy et al. (2018) had a higher Ca(OH)₂ content (9.5%) in comparison to the BOF slag used in this study (2.2%). For the BOF slags having higher amount of hydrated oxides like Ca(OH)₂, a higher degree of carbonation may happen at lower moisture content as the moisture added would be used in dissolution of CO₂ and leaching of Ca²⁺ ions than hydration of the other minerals (Baciocchi et al. 2009).

Fig. 3(b) shows the removal of CO₂ and CH₄ at different points of time until 24 hours at L/S ratios ranging from 0.1 to 0.2 (g/g). The initial steep slopes of the curves show that the carbonation reaction was fastest in the first 5 hours followed by a relatively slower reaction rate. Free lime (CaO), Ca(OH)₂ and Ca₂SiO₄ are the most reactive mineral phases for carbonation (Huijgen et al. 2005; Bonenfant et al. 2008; Uibu et al. 2011 and Su et al. 2016). The maximum CO₂ sequestration by the BOF slag after 5 hours was nearly 35 mg/g and after 24 hours was 45 mg/g. The theoretical CO₂ sequestration capacity based on the amount of [CaO (2.2%) + Ca(OH)₂ (2.4%)] present in the slag was 32 mg/g and based on total [CaO (2.2%) + Ca(OH)₂ (2.4%)] was 93 mg/g. The theoretical CO₂ sequestration capacity was

calculated based on the molar balance of the following reactions.

$$CaO(free lime) + CO_2 \rightarrow CaCO_3 \tag{3}$$

$$Ca(OH)_2 + CO_2 \to CaCO_3 + H_2O \tag{4}$$

$$Ca_2SiO_4 + 2CO_2 \rightarrow 2CaCO_3 + SiO_2 \tag{5}$$

The CO₂ sequestered in the first 5 hours corresponds to the theoretical sequestration capacity of the BOF slag based on the amount of $[CaO + Ca(OH)_2]$ present in the BOF slag. From these results it can be hypothesized that free lime and portlandite are the first minerals to react during carbonation reactions. The CO₂ sequestration in 24 hours exceeds the theoretical capacity of $[CaO + Ca(OH)_2]$ which signifies the participation of Ca₂SiO₄ in CO₂ removal. The flatter slope of the curves after 5 hours shows slower reaction kinetics. Considering all the Ca₂SiO₄ present in the BOF slag would react with CO₂, there was only 21% carbonation conversion of Ca₂SiO₄ during the experiment. This suggests slower dissolution rate of Ca₂SiO₄ under ambient condition. The reason could be the slower reaction kinetics of Ca₂SiO₄ or the formation of a carbonate layer and silicate rims over the slag particles preventing further leaching of Ca²⁺ into the solution (Huijgen et al. 2005). Overall, the BOF slag showed 43% removal of CO₂ in 24 hours calculated based on initial and final concentration of CO₂ in the vial.

Figures 4(a) and 4(b) show the SEM images of the original and carbonated BOF slag from the batch experiment, respectively. The non-carbonated BOF slag sample showed smooth surface with particles far apart and relatively more porous surface, whereas the carbonated sample showed formation of needle like outgrowths covering the pores. These outgrowths were recognized as carbonate crystals formed from carbonation reactions (Huijgen et.al. 2005; Chang et.al. 2011).



Figure 4. SEM images of BOF slag (a) Original (b) Carbonated

CONCLUSION

The CO₂ sequestration potential of the BOF slag exposed to synthetic landfill gas (50% CO₂: 50% CH₄ (v/v)) was evaluated at different moisture conditions. The results showed that the moist BOF slag can sequester considerable amount of CO₂ under landfill gas condition. Moisture was found to be a key parameter for the carbonation reaction as the BOF slag showed negligible CO₂ sequestration in a dry state (L/S ratio of 0 g/g) whereas, a substantial increase was observed in the moist state (L/S ratio of 0.1 to 0.2 g/g). In the moist state, the dissolution rates of the minerals present in the BOF slag play an important role in CO₂ sequestration. CO₂ sequestration by BOF

slag at different moisture content involves interplay between various minerals and their reaction mechanisms which needs to be confirmed with further detailed study on the dissolution characteristics as well as the reaction kinetics of the minerals present in the BOF slag. The study suggests that the BOF slag can be a potential alternative for landfill cover material to partially mitigate fugitive landfill CO₂ emission.

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Synthesis of Friedel's Salt for Application in Halide Sequestration Using Paste Encapsulation Technology

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ABSTRACT

An emerging strategy for the management of flue gas desulfurization wastewater (FGDWW) involves volume reduction of the FGDWW by concentrating to brine and then mixing the brine with fly ash and other chemical additives to form a paste suitable for landfill disposal. This process, termed as paste encapsulation technology, has not achieved the same level of success in immobilizing halides as achieved in sequestering heavy metals, because of the high solubility and elevated concentrations of halides in the brines. The focus of this study is to synthesize and characterize AFm (alumina, ferric oxide, monosulfate) phases like Friedel's salt (FS) in the laboratory using reagent-grade chemicals. The formation of these AFm phases in the brine-fly ash paste can enhance the encapsulation of halides in the paste. The structure and morphology of the synthesized samples were examined using thermogravimetric analysis (TGA), and x-ray diffraction (XRD). The total mass loss of FS, calculated using TGA, was comparable to the theoretical value. The XRD patterns also showed good agreement to a monoclinic layered double-hydroxide structure. Using these optimized operating variables, preliminary results of phase formation will be assessed using field materials such as fly ash, wastewater brine, and chemical binders.

INTRODUCTION AND BACKGROUND

Disposal of wastewater from wet flue gas desulfurization (FGD) systems is a major concern for the coal-fired power plant industry. According to a USEPA report, most of the high concentrations of metals (Ca, Na, As, Cr) and halides (Cl, Br) encountered in waste from coalfired power plants occur in FGD systems and hydraulic coal fly ash (CFA) transportation systems. The major source of halides is from coal, while additives like CaCl₂ and CaBr₂, which are added as dust suppressants and emission control measures to increase mercury capture, are minor sources. Flue gas in the wet FGD system are sprayed into or bubbled through Ca or Ca-Mg slurries in order to remove sulfur oxides (SO_x). However, along with the SO_x removal, metals and halides partition from the gas phase (flue gas) to the aqueous phase and accumulate in the FGD slurry. FGD wastewater generally contains high concentrations of total dissolved solids (TDS). Chloride (Cl) and sulfate (SO₄) are the dominant anions in FGD wastewater. Calcium (Ca), Magnesium (Mg) and Sodium (Na) are the dominant cations in the wastewater. FGD

wastewater also has significant concentrations of heavy metals like Arsenic (As), Selenium (Se) and Mercury (Hg) (USEPA 2009).

Discharge of high TDS wastewaters can have negative environmental impacts including, promotion of pitting corrosion, increased metal leaching from sediments, and formation of disinfection byproducts (Boelter et al., 1992). Therefore, USEPA released the proposed final revision to the Steam Electric Power Effluent Limitation Guidelines (ELG) which regulate FGD wastewater and other processed water (USEPA 2015), including discharge limits for many pollutants. One potential pathway being explored to manage FGD wastewater is the zero-liquid discharge (ZLD) strategy. The principle of the ZLD strategy involves reducing the volume of FGD wastewater to concentrated brine using technologies such as evaporators or advanced membrane processes such as dynamic shear enhanced membrane filtration (DSEMF), forward osmosis (FO), and membrane distillation (MD). However, such volume reduction approaches still result in a residual brine or salt that must be managed. Through the process, termed as "paste encapsulation technology", brine is mixed in cementitious matrices of fly ash and hydraulic chemical binder to form a solidified and stabilized paste. While this approach provides a material that is suitable for landfill disposal, its primary goal is to provide for long term sequestration of constituents from the FGD wastewater and coal fly ash.

Paste encapsulation technology is similar to the solidification/stabilization (S/S) typically used to treat and remediate hazardous wastes into an environmentally acceptable form. The USEPA has identified S/S as the best demonstrated available technology for 57 hazardous sites (Shi and Spence 2004). Traditional applications of S/S use ordinary Portland cement (OPC) as the primary binder (Batchelor 2006) and has been shown to be a viable treatment process for sequestrating heavy metals in the solid form (Batchelor 2006). However, the availability of coal fly ash (CFA) at facilities producing FGD wastewater provides a unique opportunity. According to a report by the American Coal Ash Association in 2015, nearly 50 million tons of fly ash are produced annually in the United States, with similar amounts produced in western Europe and throughout the world. Most types of fly ashes are considered to be pozzolonic in nature, and its geotechnical properties and lime-binding capacity makes it suitable for use in the solidification and stabilization of FGD wastewater (Ahmaruzzaman 2010). Traditional S/S approaches involve transporting the mixed material by truck and conveyor to the landfill. However, this approach has several drawbacks. The liquid-to-solid ratio is limited by what can be safely transported by truck (typically 15-18%). This range of moisture content limits the magnitude of hydration and falls short of the adequate moisture required to form stable mineral phases in the cementitious mixture.



Figure 1. Schematic representation of Friedel's salt structure (Li et al. 2017).

However, the paste technology, which transports material via pipeline, allows material to be pumped directly into a landfill. This technology allows for increase of the liquid-to-solid ratio, which means higher volumes of wastewater can be disposed with the fly ash as well as providing enough moisture to allow adequate hydration reactions. Pastes mixture can contain 30% or more