



Accelerated carbonation using municipal solid waste incinerator bottom ash and cold-rolling wastewater: Performance evaluation and reaction kinetics



E-E Chang^a, Shu-Yuan Pan^b, Liuhanzi Yang^c, Yi-Hung Chen^d, Hyunook Kim^e, Pen-Chi Chiang^{b,*}

^a Department of Biochemistry, Taipei Medical University, 250 Wu-Hsing Street, Taipei City, Taiwan 110, Taiwan, ROC

^b Graduate Institute of Environmental Engineering, National Taiwan University, 71 Chou-Shan Rd., Taipei City, Taiwan 10673, Taiwan, ROC

^c School of Environment, Tsinghua University, Haidin District, Beijing 100084, China

^d Department of Chemical Engineering and Biotechnology, National Taipei University of Technology, 1, Sec. 3, Zhongxiao E. Rd., Taipei City, Taiwan 10608, Taiwan, ROC

^e Department of Energy and Environmental System Engineering, University of Seoul, Republic of Korea

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ABSTRACT

Accelerated carbonation of alkaline wastes including municipal solid waste incinerator bottom ash (MSWI-BA) and the cold-rolling wastewater (CRW) was investigated for carbon dioxide (CO₂) fixation under different operating conditions, i.e., reaction time, CO₂ concentration, liquid-to-solid ratio, particle size, and CO₂ flow rate. The MSWI-BA before and after carbonation process were analyzed by the thermogravimetry and differential scanning calorimetry, X-ray diffraction, and scanning electron microscopy equipped with energy dispersive X-ray spectroscopy. The MSWI-BA exhibits a high carbonation conversion of 90.7%, corresponding to a CO₂ fixation capacity of 102 g per kg of ash. Meanwhile, the carbonation kinetics was evaluated by the shrinking core model. In addition, the effect of different operating parameters on carbonation conversion of MSWI-BA was statistically evaluated by response surface methodology (RSM) using experimental data to predict the maximum carbonation conversion. Furthermore, the amount of CO₂ reduction and energy consumption for operating the proposed process in refuse incinerator were estimated.

Capsule abstract: CO₂ fixation process by alkaline wastes including bottom ash and cold-rolling wastewater was developed, which should be a viable method due to high conversion.

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1. Introduction

Accelerated carbonation is an effective method for stabilizing alkaline wastes; meanwhile, permanently storing CO₂ as solid carbonates (Bobicki et al., 2012; Lackner et al., 1995; Pan et al., 2013a). In many countries such as France and Canada, natural ageing and/or ambient air carbonation are the standard practice to stabilize alkaline solid wastes such as bottom ash (Assamoi and Lawryshyn, 2012; Rendek et al., 2006; Santos et al., 2013a). CO₂ may react with metal-oxide-bearing materials to form stable and insoluble carbonates, with calcium oxide (CaO) and magnesium oxide (MgO) being the most favorable metal oxides in reacting with CO₂. Since mineral carbonation is an exothermic process, additional heat inputs and energy costs are minimal (Eloneva et al., 2008). The mineral carbonation process for CO₂ fixation was initially aimed at natural silicates (Lackner et al., 1995).

Recently, carbonation of alkaline wastes such as municipal solid waste (MSW) and steelmaking slag has been receiving more attention due to their availability, low cost, and high CaO and/or MgO contents (Bobicki et al., 2012; Pan et al., 2012). In addition, the carbonation of alkaline solids wastes can improve the chemical and physical characteristics of wastes and facilitate their reuse in a variety of applications, such as synthesis of construction materials (Fernandez Bertos et al., 2004a).

Incineration is one of the environmental friendly methods to dispose MSW, especially where recycling or reuse are not possible, because the mass and volume reductions of original waste can be up to 85% and 95%, respectively. Despite the substantial volume reduction of MSW by incineration, the MSW incinerator (MSWI) residues, i.e., bottom ashes (BA) and air pollution control (APC) ashes would be up to a mass fraction of 20% from the original MSW (Bobicki et al., 2012; Rendek et al., 2006). The MSWI-BA accounts for 80–90% of the total mass of the MSWI residues (Arickx et al., 2006). The elemental composition of MSWI-BA depends primarily on the composition of the waste input, which

* Corresponding author. Tel.: +886 2 2362 2510; fax: +886 2 2366 1642.

E-mail address: pcchiang@ntu.edu.tw (P.-C. Chiang).

Nomenclature

Materials

CRW cold-rolling wastewater
MSWI-BA municipal solid waste incinerator bottom ash

Analytical methods and models

DTG differential thermo-gravimetric analysis
DSC differential scanning calorimetric analysis
EDX energy dispersive X-ray spectroscopy

PSD particle size distribution
RSM response surface methodology
SCM shrinking core model
SEM scanning electron microscope
TGA thermo-gravimetric analysis
XRF X-ray fluorescence
XRD X-ray diffraction

may vary with location, season and recycling schemes in operation (Fernandez Bertos et al., 2004a; Teir, 2008). Since MSWI-BA is typically classified as a non-hazardous waste according to the European Waste Catalogue (European Union, 2000), it is currently being utilized as an aggregate substitute in road bases and bituminous pavement in European countries (Astrup et al., 2006; Teir, 2008).

MSWI-BA is a heterogeneous mixture of slag including ferrous and non-ferrous metals, ceramics, and other non-combustible materials. The major elements in MSWI-BA are O, Cl, Ca, Si, Al, Fe, Na, K, Mg, and C, with trace elements including Cu, Zn, S, Pb, Cr, Ni, Sn, Mn, Sb, V, and Co (Teir, 2008; Todorovic and Ecke, 2006). Mineralogical studies indicate that the main crystalline phases of MSWI-BA are typically silicates (e.g., quartz [SiO₂], gehlenite [Ca₂Al₂SiO₇], olivine [(Mg, Fe)₂SiO₄], and augite [(Ca,Na)(Mg,Fe,Al,Ti)(Si,Al)₂O₆]), sulfates (e.g., anhydrite [CaSO₄], ettringite [Ca₆Al₂(SO₄)₃(OH)], and gypsum [CaSO₄·2H₂O]), carbonates (e.g., calcite [CaCO₃]), and hematite [Fe₂O₃] (Fernandez Bertos et al., 2004a; Teir, 2008). With the specific chemical characterizations of MSWI-BA, it exhibits its potential CO₂ fixation capability due to its calcium content and alkaline properties. In addition, the mineral carbonation of MSWI-BA can immobilize heavy metals and effectively prevent their leaching, especially for Cr, Cu, Pb, Zn, and Sb (Arickx et al., 2006; Fernandez Bertos et al., 2004a; Todorovic and Ecke, 2006).

Mineral carbonation processes conducted in the aqueous phase have proven to be more effective than those utilizing the dry gas–solid method (Bobicki et al., 2012; Fernandez Bertos et al., 2004g; Pan et al., 2012). In addition, aqueous carbonation of industrial solid wastes can be coupled with alkaline wastewater resulting in further lowering material and energy consumptions (Chang et al., 2013; Pan et al., 2013a). Therefore, the objectives of this study were (1) to evaluate the performance of aqueous carbonation using MSWI-BA coupled with cold-rolling wastewater (CRW) via a slurry reactor for CO₂ fixation, (2) to determine the reaction kinetics and the rate-limiting steps of accelerated carbonation based on the shrinking core model (SCM), (3) to establish a response surface model for visualizing the effect of different operating parameters on carbonation conversion, and (4) to estimate the energy consumption and operating cost of the developed process for CO₂ fixation.

2. Materials and methods

2.1. Materials

The ground MSWI-BA provided by Bali Refuse Incineration was sieved into three categories. i.e., <125 μm, 125–350 μm, and 350–500 μm, and dried at 105 °C for 8 h to eliminate moisture. After that, the MSWI-BA was stored at room temperature in small airtight containers and then placed in a larger capped container. In addition, high-pressure CO₂ gas with a volumetric concentration

of 99% was supplied by Ch'ing-Feng Gas Corporation (Taipei, Taiwan).

On the other hand, CRW produced from a steel manufacturer (Company B) was used as the liquid agent during the carbonation reaction in a slurry reactor. The CRW is the inevitable wastewater from cold rolling process in steelmaking industry, which typically needs additional neutralization and treatment before its discharge from the industry. Generally, the characteristics of CRW can be classified into three groups: alkaline (inorganic), acidic (inorganic), and oily (organic). In this study, the alkaline CRW was used (i.e., pH ~ 11.3), containing sodium of 800–1000 mg/L, potassium of 150–270 mg/L, chloride of 1400 mg/L, and sulfate of 150–230 mg/L.

2.2. Aqueous carbonation experiments

The effects of the operating conditions including reaction time, liquid-to-solid (L/S) ratio, gas flow rate, and particle size on the carbonation conversion of MSWI-BA were evaluated. Fig. 1 shows a

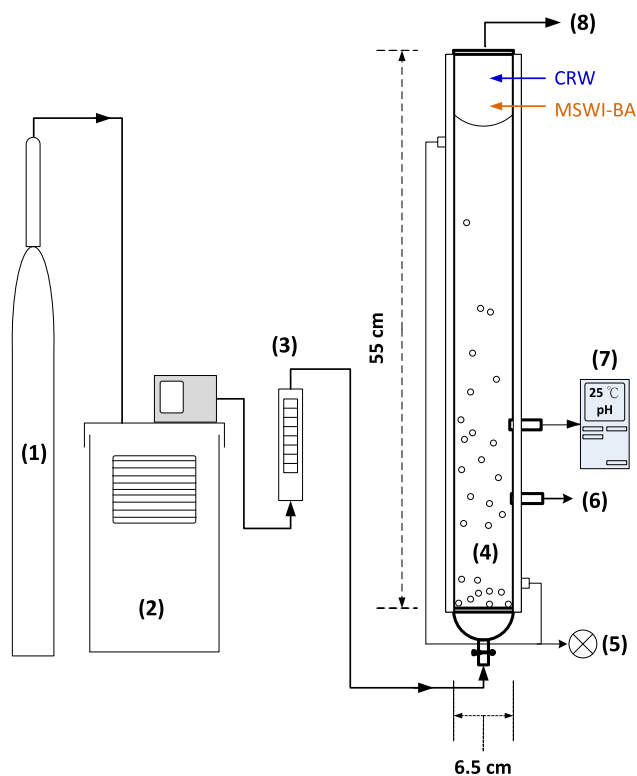


Fig. 1. Schematic diagram of the experimental set-up for carbonation of MSWI-BA in a slurry reactor. (1) CO₂ gas cylinder; (2) circulating bath; (3) rotameter; (4) slurry reactor; (5) heating jacket; (6) sampling; (7) thermo couple and pH analyzer; (8) vent to hood.

schematic diagram of the carbonation experiments for MSWI-BA with CRW in a slurry reactor. The slurry reactor was a glass column of 0.55 m in height and 0.065 m in diameter, and the sampling port was located at 0.02 m above the bottom of the reactor. All experiments were carried out in batch mode with CRW and MSWI-BA under various levels of L/S ratio, i.e., 5, 10, 20, and 40 mL/g. CO₂ was injected from the bottom of the reactor continuously at approximately 1 atm and at different flow rates, i.e., 0.2, 0.5, 1.0, and 1.5 L/min. All experiments were conducted with a reaction time of 120 min, and the reacted MSWI-BA slurry was sampled at 1, 5, 10, 30, 60, and 120 min.

2.3. Carbonation conversion of MSWI-BA

The chemical properties of MSWI-BA were measured in accordance with ASTM method C114 using X-ray fluorescence (XRF, PW2430, Philips, Netherland). The densities and BET surface areas of the MSWI-BA samples were analyzed by a pycnometer (Accupyc 1340, Micromeritics Instrument Corporation, USA) and a low-temperature N₂-adsorption BET apparatus (ASAP2010, Micromeritics Instrument Corporation, USA), respectively. The particle size distribution (PSD) of the MSWI-BA was obtained by laser diffraction (Beckman Coulter LS 230), which was adapted from the ISO 13320:2009 method (International Organization for Standardization, 2009).

The carbonation conversion of the MSWI-BA before and after carbonation reaction were examined by a thermogravimetric and differential scanning calorimetry (TG–DSC, STA6000, PerkinElmer, USA), which determines the sample weight loss at different temperatures as well as the phase transformation of sample. Since the iron-bearing compounds were contained in relatively low contents in fresh MSWI-BA and generally combined with aluminate and silicate in the solid matrix, both the leaching rate and concentration of iron were expected to be limited (Pan et al., 2013a). As a result, the formation of iron carbonate could be negligible due to its very low contents. Therefore, it suggests that the calcium-bearing compound should be one of the major chemical components in the accelerated carbonation reaction.

Approximately 15–25 mg aliquots of the MSWI-BA samples were heated linearly in TGA at the temperature ranges between

50 and 900 °C under a heating rate of 10 °C/min and an N₂ flow rate of 20 mL/min. Because calcium carbonate (CaCO₃) will typically decompose into calcium oxide (CaO) and CO₂ within the temperature range between 500 and 850 °C, the amounts of CO₂ released from dry mass of MSWI-BA estimated by the weight loss (Δm_{CO_2}), in the above temperature ranges can be calculated by Eq. (1):

$$\text{CO}_2 \text{ (wt.\%)} = \frac{\Delta m_{\text{CO}_2}}{m_{105\text{ }^\circ\text{C}} - \Delta m_{\text{CO}_2}} \times 100\% \quad (1)$$

where $m_{105\text{ }^\circ\text{C}}$ is the dry weight of MSWI-BA sample. With the above calculation, the carbonation conversion of MSWI-BA (δ_{CaO} (%)), defined as the amount of CO₂ actually captured in dry mass of each sample compared with the calculated theoretical extent of carbonation based on the reactive-oxide content of MSWI-BA can be determined using the following equation (Eq. (2)):

$$\delta_{\text{CaO}} \text{ (\%)} = \frac{\text{CO}_2 \text{ (wt.\%)} \times \frac{\text{MW}_{\text{CaO}} \text{ (g/mole)}}{\text{MW}_{\text{CO}_2} \text{ (g/mole)}}}{\text{CaO}_{\text{total}} \text{ (g/g)}} \quad (2)$$

where MW_{CO₂} (g/mole) is the molecular weight of CO₂; MW_{CaO} (g/mole) is the molecular weights of CaO; and CaO_{total} is the percent weight fraction of CaO (mass/mass) in the fresh sample.

2.4. Characterization of MSWI-BA using SEM, EDX and XRD

Morphological investigations of fresh and carbonated MSWI-BA were performed with a scanning electron microscope (SEM, JSM-4500, JEOL, Japan). The samples before and after carbonation were mounted with double-sided carbon tape on an aluminum stub, and then coated with a thin-layer of carbon. The accelerating voltage was set at 10 kV. In addition, the distributions of elements in the fresh and carbonated MSWI-BA were determined using energy dispersive X-ray spectroscopy (EDX, JED-2300, JOEL, Japan), which is useful for identifying CaCO₃ formed on the surface of the MSWI-BA during the carbonation reaction.

The mineralogy of MSWI-BA before and after carbonation was qualitatively identified and characterized by X-ray diffraction (XRD, Bruker D8 Advance). The X-ray source was a Cu anode operating at 40 kV and 40 mA using Cu K α radiation with a diffracted beam graphite-monochromator. Since the major mineral phases

Table 1
Summary of physicochemical properties of MSWI-BA used in this study.

Items		Fresh MSWI-BA			
		<125 μm	125–350 μm	350–500 μm	
Physical properties	True density ^a (g cm ⁻³)	2.70	2.73	2.78	
	Mean diameter (μm)	59.6	175.8	501.5	
	Median diameter (μm)	45.1	164.9	513.5	
	BET surface area ^a (m ² g ⁻¹)	6.63 \pm 0.02	4.00 \pm 0.02	3.51 \pm 0.01	
	Langmuir surface area ¹ (m ² g ⁻¹)	9.31 \pm 0.30	5.57 \pm 0.20	4.87 \pm 0.17	
Chemical properties	XRF	SiO ₂ (%)	44.9	50.7	45.0
		CaO (%)	21.1	16.4	18.3
		Al ₂ O ₃ (%)	9.3	8.7	9.5
		Fe ₂ O ₃ (%)	8.5	9.3	11.8
		Na ₂ O (%)	5.0	5.9	5.2
		P ₂ O ₅ (%)	2.6	2.3	2.6
		MgO (%)	2.1	1.9	2.1
		TiO ₂ (%)	1.4	1.2	1.4
		SO ₃ (%)	1.3	0.8	0.9
		K ₂ O (%)	0.9	0.8	0.8
		CaCO ₃ (%)	7.07 \pm 0.32	2.57 \pm 0.02	3.38 \pm 0.55
		f-CaO (%)	0.07	0.00	0.07
		TGA			
	Chem. ^b				
Theoretical CO ₂ capture capacity (g CO ₂ /kg BA) ^c		126.9	112.7	123.8	

^a Analyzed by Particulate Technology Laboratory, NTU.

^b Chemical analysis.

^c Estimated by Eq. (3).

in MSWI-BA can be identified within between 20° and 80° in 2θ , data collection was carried out between the angel range with a scan step of 0.008° and count time of 0.5 s per step.

3. Results and discussion

3.1. Performance evaluation of carbonation using MSWI-BA and CRW

Table 1 summarizes the physico-chemical properties of the MSWI-BA with various particle sizes used in the laboratory. The MSWI-BA was ground and sieved into three categories, i.e., <125 μm , 125–350 μm , and 350–500 μm , with the BET surface area of 6.63, 4.00, and 3.51 m^2/g , respectively. The major components in the MSWI-BA were SiO_2 , CaO , Al_2O_3 , and Fe_2O_3 , while the trace compounds were Na_2O , MgO , P_2O_5 , TiO_2 , and SO_3 . Among the metal oxides, the CaO content was the highest within the MSWI-BA (i.e., 21% of the particles with the size of less than 125 μm). The mineralogical characteristics of MSWI-BA before and after the carbonation were determined by XRD and presented in Fig. 3(a) and (b), respectively. The results show that the mineral composition of fresh MSWI-BA included quartz (SiO_2), calcite (CaCO_3), epidote (Al-Ca-Fe-Si-O-H), tasselite (Ca-Fe-Mg-Na-P-O-H), ktenasite (Cu-H-O-S-Zn), and trace amounts of anhydrite (CaSO_4), pigeonite ($\text{Al-Ca-Fe-Mg-Mn-Ti-Na-Si-O}_2$), and gehlenite ($\text{Al-Ca-Fe-Mg-Si-O}_2$), of which quartz was the main component. Even though Ca(OH)_2 was not identified by the XRD measurements, Ca-Al-Si oxide and Ca-Na-Si oxide were present in significant quantities, as indicated by the higher peak intensities in the XRD pattern of fresh MSWI-BA. Calcium-bearing compounds in fresh MSWI-BA are primarily composed of various types of oxides with silicates that contain other metals such as Fe and Al, which are consistent with observations reported in the literature (Bobicki et al., 2012; Fernandez Bertos et al., 2004a).

In contrast to the XRD results of fresh MSWI-BA, the XRD pattern of the reaction products identified calcite (i.e., CaCO_3) as the primary component. Similar to the fresh MSWI-BA, on the other hand, quartz was found to be a major component of the carbonated MSWI-BA according to the XRD results. The XRD results also revealed that the calcium-bearing species in MSWI-BA were primarily composed of various types of silicates (e.g., CaO-Al-silicates , CaO-Fe-silicates). The dissociation of calcium-bearing compounds in the fresh MSWI-BA should occur after introducing the MSWI-BA into the CRW. The Ca^{2+} will react with carbonate ions (CO_3^{2-}) under an alkaline condition, leading to the formation of CaCO_3 upon introducing of CO_2 into the slurry reactor. Therefore, the leaching concentration of Ca^{2+} from the solid waste into the solution is an important indicator for carbonation reaction.

It was noted that the probability of MgCO_3 formation by carbonation of MSWI-BA was very low due to both the relatively low content of MgO in the MSWI-BA and the conditions of low CO_2 pressure with short reaction time (Chang et al., 2012). The other metal oxide components such as SiO_2 and P_2O_5 in the fresh MSWI-BA were also considered not to contribute to CO_2 fixation. Therefore, only calcium species were considered as the major components that participate in the carbonation reaction. The theoretical amounts of CO_2 captured in the fresh MSWI-BA can be calculated as Eq. (3) (Huntzinger et al., 2009; Steinour, 1959):

$$\text{ThCO}_2 = \frac{44}{56} \left(\text{CaO} - \frac{56}{100} \times \text{CaCO}_3 - \frac{56}{80} \times \text{SO}_3 \right) \quad (3)$$

where ThCO_2 ($\text{g CO}_2/\text{g MSWI-BA}$) is the theoretical CO_2 capture capacity, CaO (g CaO/g MSWI-BA) and SO_3 ($\text{g SO}_3/\text{g MSWI-BA}$) are the weight fraction of CaO and SO_3 in MSWI-BA measured by XRF, respectively, and CaCO_3 ($\text{g CaCO}_3/\text{g MSWI-BA}$) is the weight fraction of CaCO_3 analyzed by TGA.

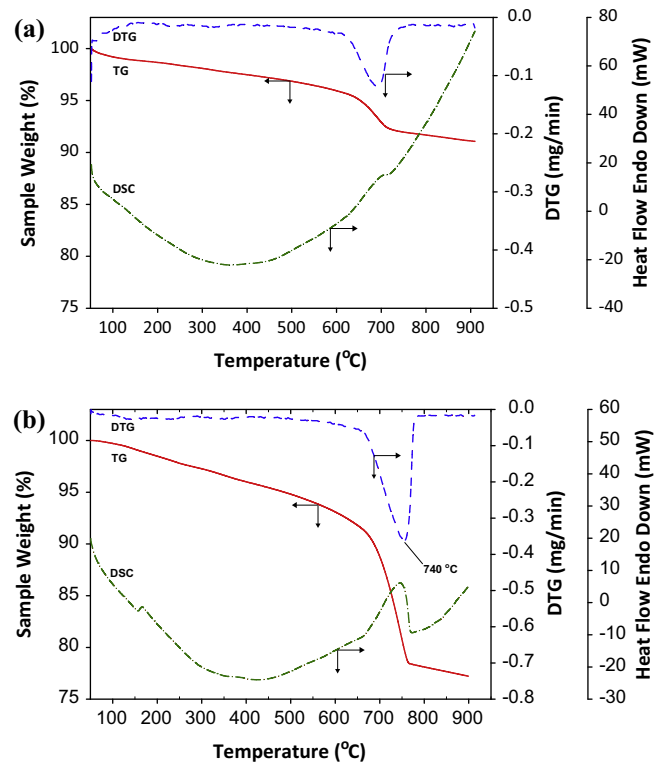


Fig. 2. TG–DTG curves of (a) fresh and (b) carbonated MSWI-BA ($P_{\text{CO}_2} = 14.7$ psia; $Q_{\text{CO}_2} = 0.5$ L/min; $T = 25$ °C; $t = 120$ min; $D_p = 125$ – 350 μm ; L/S ratio = 10:1 mL/g; carbonation conversion = 90.7%).

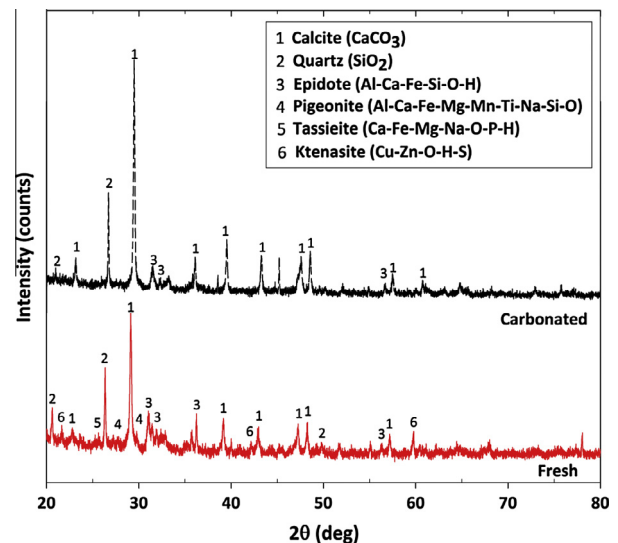


Fig. 3. X-ray diffraction (XRD) pattern of fresh and carbonated MSWI-BA.

According to the chemical analysis shown in Table 1, the theoretical CO_2 fixation capacity of the MSWI-BA was estimated to be 127 g CO_2 per kg of MSWI-BA for a particle size of less than 125 μm . The CO_2 fixation capacity was validated by the TGA measurements as presented in Fig. 2(a) and (b), which shows that the TG–DTG curves of the fresh and carbonated MSWI-BA, respectively. The content of CaCO_3 in the fresh MSWI-BA was 7.1% in dry weight, which corresponds to the weight loss within the temperature ranges between 600 and 750 °C (Fig. 2(a)). After carbonation, the CaCO_3 content significantly increased with a DTG peak at

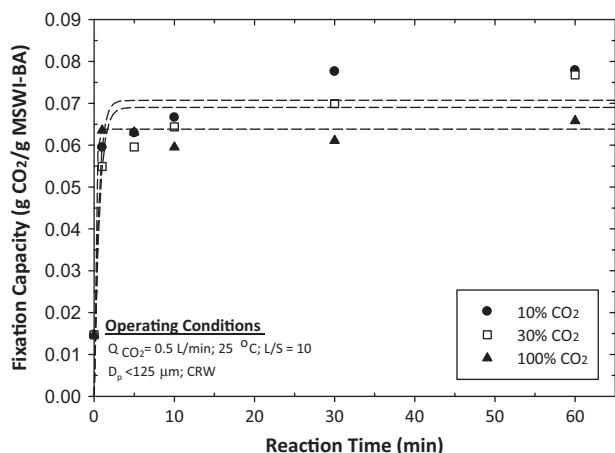


Fig. 4. Influence of CO₂ concentration on capture capacity of MSWI-BA via a slurry reactor (carbonation conditions: L/S ratio = 10:1 mL/g; Q_{CO₂} = 0.5 L/min; P_{CO₂} = 14.7 psia; T = 25 °C).

740 °C. In addition, the content of CaO shown in Table 1 was not attributed to the Ca(OH)₂ because a DTG peak was not observed at temperatures of 300–500 °C.

In addition, the effect of CO₂ concentration on the fixation capacity of MSWI-BA was evaluated (Fig. 4); three different CO₂ concentrations (i.e., 100%, 30%, and 10%) were applied. Since the volume of MSWI-BA slurry was fixed for every experiment, the carbonation conversion of MSWI-BA was higher with a lower CO₂ concentration (e.g., 10% CO₂) due to the longer duration time for remaining higher alkalinity and carbonate concentration in solution. In the case of the CO₂ concentration of 10%, which is similar to concentration of CO₂ in the flue gas from an incinerator, the fixation capacity of MSWI-BA was found to be 0.078 kg CO₂ per kg of ash for 30 min in a slurry reactor. According to the statistics reported by environmental protection administration (EPA) of Taiwan, the annual production of bottom ash from the Bali incinerators was estimated to be 83,100 ton in 2013, as shown in Table 2.

Table 2

Information of Bali Refuse Incineration Plant and its statistics on waste treatment and power recovery in 2013.

Categories	Items	Unit	Values
Specification	Area of plant	Ha	3.5
	Number of incinerators	–	3
	Designed capacity of incineration disposal	tons/day	1350
Treatment statistics	Water content of refuse received	%	35–55
	Refuse received	tons	387,658
	Refuse incinerated	tons	383,110
	Ashes from incinerator	tons	83,100
Heat/power recovery	Installation capacity of electric power generation	kW	35,771
	Electric power generation	MW h	234,590
	Percentage of power sold	%	79.70
Air pollutant emissions ^a	Total CO ₂ emissions	tons	290,000
	Dioxin	ng-TEQ/Nm ³	0.038–0.043 (0.1 ^b)
	SO ₂ (daily average)	ppm	0.003–0.005 (0.1 ^b)
	CO (hour max.)	ppm	0.5–0.7 (35 ^b)
	NO _x (daily average)	ppm	0.026–0.01
	THC (daily average)	ppm	2.9–3.4
	TSP per day	μg/m ³	34–78 (250 ^b)

^a Average from Jan. to May in 2014.

^b Regulated concentration.

Therefore, the annual CO₂ emission from the Bali incinerators could be expected to reduce by 6480 ton per year, corresponding to a 2.24% reduction in total annual CO₂ emission (i.e., 290,000 ton), using the MSWI-BA carbonation process developed in this study.

It was noted that the CRW could enhance the leaching of metal ions (e.g., Ca²⁺) from the solid waste into the solution (Chang et al., 2013; Pan et al., 2013a, 2013b). According to the quantitative X-ray diffraction (XRD) results reported by Pan et al. (2013b), the calcium-silicate-bearing parts in alkaline solid waste might react with the inorganic ionic species (e.g., Na⁺ and Cl⁻) in the CRW by formation of surface complexes. Therefore, the leaching of Ca²⁺ can be promoted because of the reductive and/or oxidative dissolution of minerals. In this study, the chemical properties of CRW were found to be improved after carbonation process, as presented in Table 3. For example, the CRW was slightly alkaline (i.e., pH ~ 8.9) before introduced to the carbonation process whereas the pH value of CRW dropped to ~6.3 after the carbonation. In addition, the concentrations of several metal ions including aluminate and iron were reduced after the carbonation because the chemical precipitation occurred. However, the amounts of those metal carbonates were too low to be identified by XRD. Nonetheless, the CRW after the carbonation process should be treated prior to its final disposal, where the conventional industrial wastewater treatment processes can be readily applied for this purpose.

The proposed carbonation process can not only improve the chemical properties of alkaline wastes but also reduce the CO₂ emission from incinerators by co-utilizing the CRW and MSWI-BA to fix CO₂ in the flue gas via the slurry reactor. It was also noted that a carbonation conversion of >85% for solid wastes should be acceptable to achieve waste stabilization and CO₂ fixation (Pan et al., 2015). Therefore, both economic and environmental benefits can be obtained by reducing the treatment cost of wastewater and increasing the added-value of bottom ash for construction materials through the application of the proposed CCU technology. In addition, the amount of CO₂ reduction can be considered as the certified emission reduction (CER) credits, which is beneficial to the emission trading scheme (ETS) under the clean development mechanism (CDM) issued by the Kyoto Protocol.

3.2. Kinetic model of carbonation for MSWI-BA

Fig. 5 presents the influence of reaction time on the carbonation conversion of MSWI-BA with different particle sizes. The experimental results revealed a rapid reaction rate at the beginning of reaction (i.e., up to approximate 5 min) because of the high pH value of slurry, which is beneficial to the formation of CO₃²⁻ ions. Along with the CO₂ dissolution and uptake, the H⁺ ions were formed continuously into the solution which leads to a decrease of reactant concentrations, thereby lowering the reaction rate. Therefore, the carbonation rate decreased gradually as reaction time increased, which were consistent with those reported in the literature (Arickx et al., 2006; Fernandez Bertos et al., 2004a).

In this investigation, experimental data were utilized to determine the reaction kinetics and rate-determining step of the carbonation, as well as the effective diffusion coefficients based on the shrinking core model (SCM). The possible rate-determining steps include (1) fluid-film diffusion, (2) ash-layer diffusion, and (3) chemical reaction at the un-reacted core surface (Levenspiel, 1999). The carbonation reaction first occurs at the outer layer of the MSWI-BA and then proceeds at a narrow front, which moves into the MSWI-BA and leaves behind reaction product (i.e., CaCO₃) and reactive-species-depleted rims referred to as the “ash” layer. Therefore, an un-reacted core of material gradually shrinks in size during the reaction (Levenspiel, 1999).

Table 3
Physico-chemical properties of CRW before and after carbonation process^a.

Water quality parameters		Unit	Fresh CRW	MSWI + CRW (0 min)	Treated CRW (120 min)
pH		–	8.9 ± 0.1	12.0 ± 0.2	7.5 ± 0.1
Temp		°C	24.9 ± 0.02	25.0 ± 0.02	23.4 ± 0.04
Conductivity		µmho/cm	6700 ± 30	6400 ± 130	3510 ± 10
TDS		mg/L	3720 ± 15	3200 ± 35	1190 ± 30
Anion concn. ^b	F ⁻	mg/L	0.98 ± 0.02	0.95 ± 0.05	0.59 ± 0.03
	Cl ⁻	mg/L	1780 ± 422	1900 ± 126	389 ± 35
	NO ₃ ⁻ N ⁻	mg/L	N.D. (<0.036)	0.92 ± 0.04	1.25 ± 0.04
	SO ₄ ²⁻	mg/L	196 ± 37	190 ± 15	440 ± 18
	PO ₄ ³⁻	mg/L	N.D. (<0.065)	N.D. (<0.065)	N.D. (<0.065)
Cation concn. ^c	K ⁺	mg/L	260 ± 87	115 ± 19	202 ± 21
	Na ⁺	mg/L	843 ± 14	405 ± 21	615 ± 16
	Ca ²⁺	mg/L	8.82 ± 3.87	85.5 ± 4.4	52.8 ± 5.2
	Mg ²⁺	mg/L	0.70 ± 0.46	0.53 ± 0.16	6.55 ± 1.12
	Al ³⁺	mg/L	N.D. (<0.001)	7.29 ± 0.39	0.28 ± 0.02
	Fe ³⁺	mg/L	1.21 ± 0.65	0.04 ± 0.01	0.02 ± 0.01
	Cr ³⁺	mg/L	N.D. (<0.001)	0.13 ± 0.01	0.22 ± 0.01
	Cu ²⁺	mg/L	N.D. (<0.001)	0.05 ± 0.01	0.31 ± 0.03
	Li ⁺	mg/L	0.002 ± 0.001	0.009 ± 0.001	0.028 ± 0.006
	Mn ²⁺	mg/L	N.D. (<0.001)	0.002 ± 0.001	0.149 ± 0.018
	Ni ²⁺	mg/L	0.002 ± 0.001	0.002 ± 0.001	0.014 ± 0.001

^a A total of three replicates were analyzed.

^b The anion concentrations were determined by IC (Dionex DX-100, Thermo, USA). The concentration of Cl⁻ and SO₄²⁻ was measured by precipitation titrimetry (i.e., Mohr method) and the Nephelometer method, respectively.

^c The cation concentrations were determined by ICP-OES (710 Series, Agilent, USA).

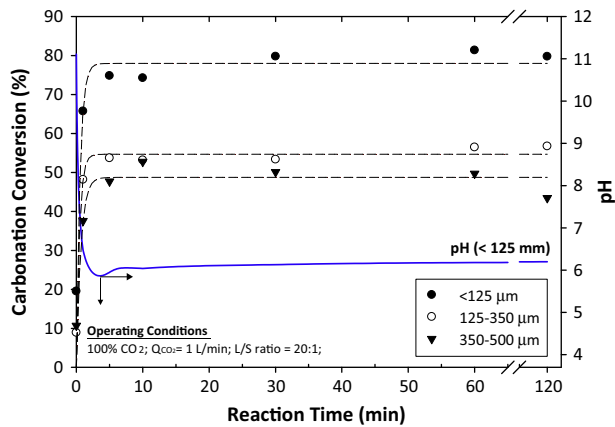


Fig. 5. Influence of reaction time and particle size on pH value and carbonation conversion of MSWI-BA in a slurry reactor (carbonation conditions: L/S ratio = 20:1 mL/g; Q_{CO_2} = 1 L/min; P_{CO_2} = 14.7 psia; T = 25 °C).

When diffusion of reactants through the ash layer is the rate-limiting step, Eq. (4) illustrates the dependence of the reaction time on carbonation conversion (X_B) and the effective diffusivity of a gas reactant in the ash layer (D_e (cm²/s)):

$$t = \frac{\rho_B R^2}{6bD_e C_{Ag}} [1 - 3(1 - X_B)^{2/3} + 2(1 - X_B)]$$

$$= \tau [1 - 3(1 - X_B)^{2/3} + 2(1 - X_B)] \quad (4)$$

where the term ρ_B (mole cm⁻³) is the molar density of the MSWI-BA, R (cm) is the average radius of the MSWI-BA, which is assumed to remain constant, C_{Ag} (mole cm⁻³) is the CO₂ concentration in the fluid, τ (s) is the time for complete conversion of a reactant particle to product, and the stoichiometric coefficient (b) is assumed to be one. More detailed discussion regarding the kinetic model of carbonation for determining the fluid-film diffusion controlled and chemical reaction controlled conversions can be found elsewhere (Chang et al., 2011, 2012; Lekakh et al., 2008; Levenspiel, 1999).

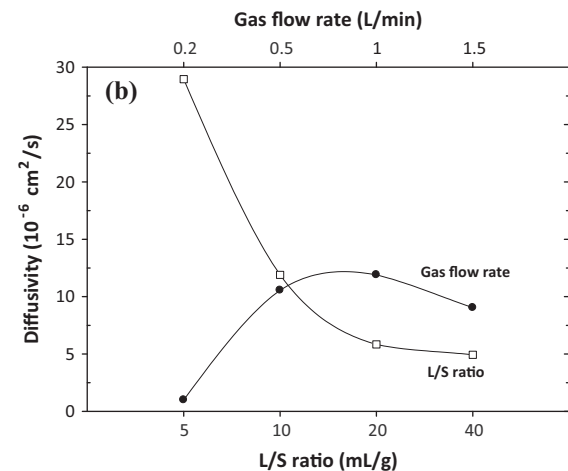
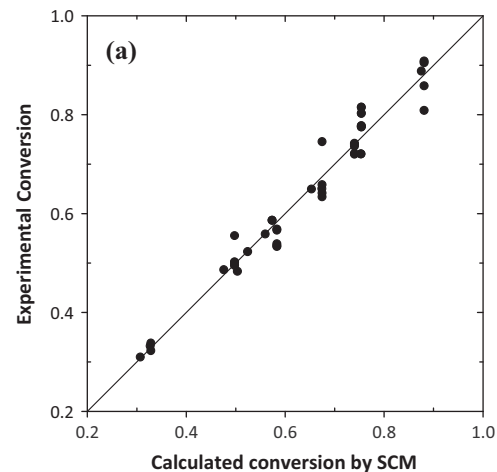


Fig. 6. (a) Results of predicted carbonation conversion of MSWI-BA by SCM with experimental data in a slurry reactor; (b) variation of diffusivity (D_e) with L/S ratio and gas flow rate for MSWI-BA carbonation based on the SCM (carbonation conditions: D_p = 125–350 µm; P_{CO_2} = 14.7 psia; T = 25 °C).

Fig. 6(a) presents the carbonation conversion of MSWI-BA predicted by the SCM along with experimental data obtained from the slurry reactor. As shown in the figure, errors of less than $\pm 10\%$ could be observed between the model prediction and experimental data. This result suggests that the carbonation conversion of MSWI-BA in the slurry reactor operated at various conditions could be well predicted by the SCM. It also suggests that the carbonation reaction of the MSWI-BA in the slurry reactor was controlled by the ash-layer diffusion mechanism because the experimental data was well fitted to Eq. (4). As shown in Fig. 6(b), the D_e values are in the range of 1.00×10^{-6} – 2.90×10^{-5} cm^2/s , which suggests that the carbonation reaction of MSWI-BA should be ash-diffusion controlled. These D_e values are relatively higher than those reported in the literature. For instance, in the study performed by Chang et al. (2012), the D_e values for carbonation of steelmaking slag with DI water in a slurry reactor were in the range of 2.88×10^{-7} – 7.28×10^{-7} cm^2/s . It suggests that the CRW could increase the leaching of calcium ions from the solid wastes, thereby enhancing the carbonation reaction.

The carbonated MSWI particles exhibited an ash layer of CaCO_3 after carbonation, which could be verified by the SEM images (Fig. 7(a) and (b)). Each MSWI-BA was observed to possess smooth surface prior to carbonation reaction. However, after carbonation, the characteristic rhombohedral or plate-like crystals were found in the SEM images provided in Fig. 7(a) and (b). Obviously, the microstructure of the MSWI-BA was modified because of accelerated carbonation, which was validated by the EDX analyses. It

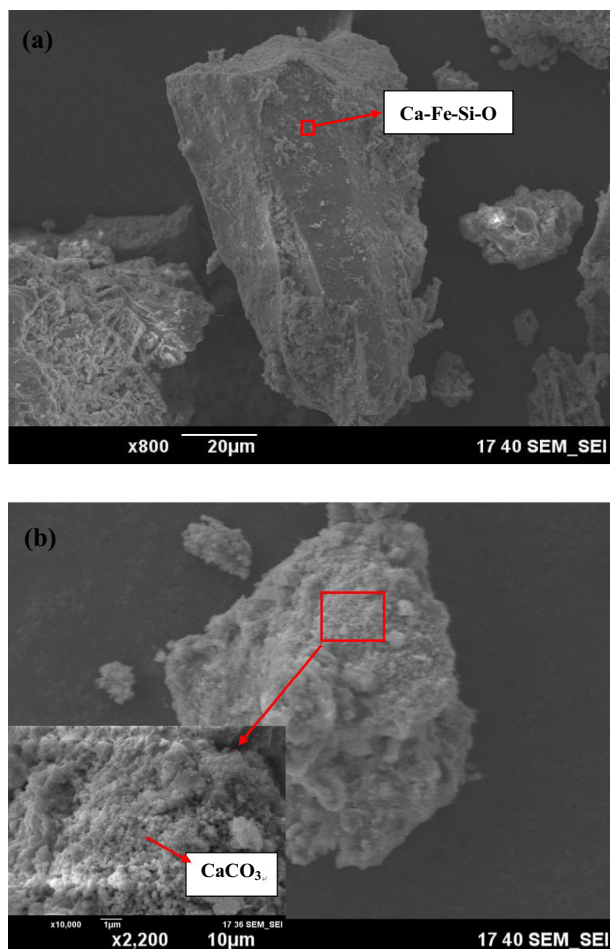


Fig. 7. Scanning electron microscope (SEM) images with EDX patterns for (a) fresh and (b) carbonated MSWI-BA.

was revealed that the carbonated MSWI-BA is composed primarily of Ca, O, and C, indicating a calcium carbonate (CaCO_3) composition. Parts of the CaCO_3 product appear to be isolated and separated, with a size of 1–3 μm and a more regular morphology. In some cases, the CaCO_3 layer was formed as compact and continuous coatings on the surface of carbonated MSWI-BA with a random structure of 1–3 μm in length. It was thus concluded that the MSWI-BA was successfully carbonated with CO_2 and formed the calcite precipitation (CaCO_3) in a slurry reactor.

3.3. Identification of key operating factors for response surface model

According to Fig. 5, it was observed that the conversion of MSWI-BA increased with the particle size decreased with an L/S ratio of 10:1. During the first 5 min of carbonation, pH of the MSWI-BA slurry decreased from 11.5 to 6.0 because of the hydrolysis of calcium-bearing compounds in MSWI-BA, which could induce a rapid uptake of OH^- ions. After introducing CO_2 gas into the reactor, carbonate ions (CO_3^{2-}), formed upon the dissolution of CO_2 in aqueous solution under alkaline conditions, and reacted with calcium ions (Ca^{2+}) to form CaCO_3 precipitation. Consequently, the system was observed to reach an equilibrium pH value of 6.35 from 5 min to 120 min, and the carbonation conversion of MSWI-BA was found to be constant. Similar results were observed in the literature (Chang et al., 2011; Pan et al., 2012, 2013b; Santos et al., 2013b).

Fig. 8(a) shows the influence of L/S ratio, ranging from 5 to 40 mL/g, on the conversion of MSWI-BA in the slurry reactor. It was observed that the conversion of MSWI-BA with an L/S ratio

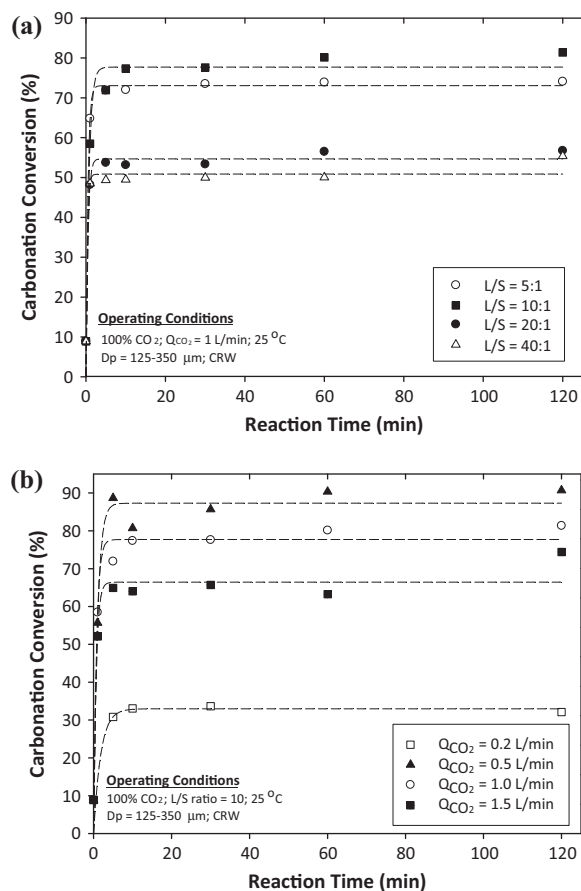


Fig. 8. Influence of (a) L/S ratio and (b) CO_2 flow rate on carbonation conversion of MSWI-BA in a slurry reactor (carbonation conditions: $D_p = 125$ – 350 μm ; $P_{\text{CO}_2} = 14.7$ psia; $T = 25$ $^\circ\text{C}$).

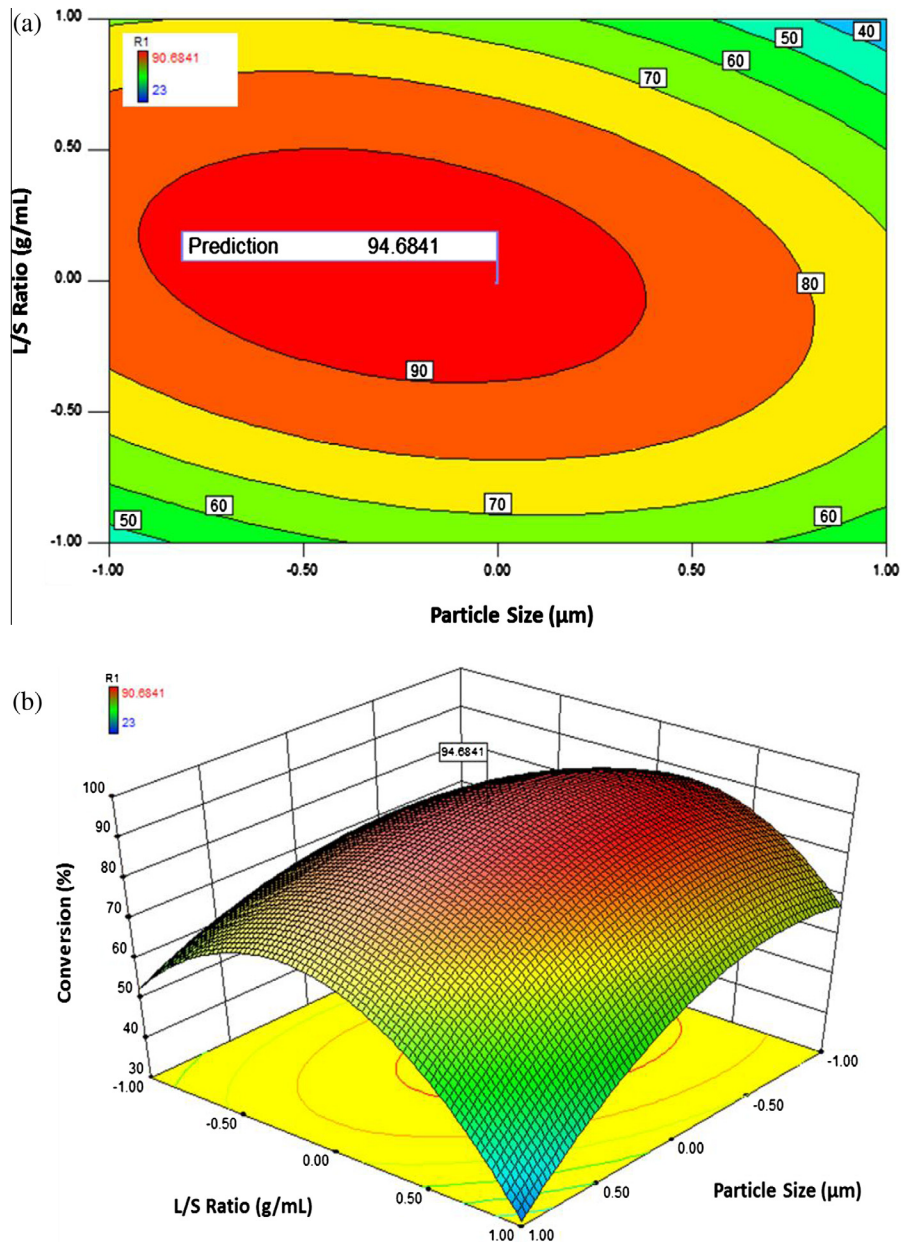


Fig. 9. (a) 2-D contour plot of response of conversion using an RSM, and (b) 3-D response surface plot of conversion response using an RSM.

of 10 mL g^{-1} was the highest one and gradually approached to a stable value of 70%. The experimental results indicate that the conversions of the MSWI-BA were lower at an L/S ratio of less than 5 mL/g , because of the slow reaction kinetics among gas, liquid, and solid phases, which reflects the natural carbonation of minerals in the environment. Therefore, it suggests that the L/S ratio should be greater than 5 mL/g to achieve a better carbonation of MSWI-BA. At an L/S ratio of greater than 10 mL/g , however, the carbonation conversion of MSWI-BA also would decrease due to the less amount of MSWI-BA available in a liquid agent, resulting in a lower concentration of calcium ions in the slurry.

Fig. 8(b) shows the influence of CO_2 gas flow rate on carbonation conversion of MSWI-BA; the L/S ratio was set at 10 mL/g . The carbonation conversion of MSWI-BA was observed to increase when the gas flow rate increased from 0.2 to 0.5 L/min and then to decrease when the gas flow rate further increased from 0.5 to 1.5 L/min . In general, increased gas flow rate is expected

to enhance the mass transfer of gaseous CO_2 into slurry solution, which would increase the carbonation conversion of MSWI-BA. However, a higher gas flow rate would result in a rapid decrease of the pH value and CO_3^{2-} concentration in the solution, which is not beneficial to carbonation. In fact, Fig. 8(b) suggests that the gas flow rate for MSWI-BA/CRW carbonation in the slurry reactor should be maintained at 0.5 L/min . In this case, a carbonation conversion of 90.7%, corresponding to a CO_2 fixation capacity of $102 \text{ g per kg MSWI-BA}$, was achieved.

Based on the above experimental works, the effect of different operating parameters on carbonation conversion of MSWI-BA was statistically evaluated by response surface methodology (RSM) using experimental data to predict the maximum carbonation conversion. Design Expert software package 8.0.6 (StatEase Inc., Minneapolis, USA) was used for regression analysis and analysis of variance (ANOVA) on the response surface. The obtained R^2 value for the response surface was 0.992, which indicates that the

Table 4Estimation of power consumption and operating cost for capturing 1 ton of CO₂ using MSWI-BA via slurry reactor.

Process	Equipment	Specification/equipment scale	Power consump. per unit process (kW h) (1)	Treated capacity (h ⁻¹) (2)	Capacity per unit process (h ⁻¹) (3)	Power consumption (kW h) (1) * (2)/(3)	Cost estimation (US\$) ^c
Grinding ^a	Crusher (ball mill)	≤125 μm	21.64	9.8 t	1 t	212.1	19.3
		125–350 μm	14.72	9.8 t	1 t	144.3	13.1
		350–500 μm	9.20	9.8 t	1 t	90.1	8.20
Blower ^b	Side channel blower	75% full load, motor nameplate efficiency: 82.1%	18.0	13,800 m ³	6900 m ³	36.0	3.28
Total						180.3	16.4

^a Estimated from Bond's equation and work index is assumed to be 30.4 kW h/ton.^b Available data was obtained and simulated from Engineering ToolBox.^c Taiwan average electricity price for industry in 2013 was US\$ 0.091 per kW h. The total cost was based on the scenario of a particle size of 125–350 μm.

experimental data can be well expressed by the response surface. The predicted model for carbonation conversion of MSWI-BA in terms of coded variables can be expressed by Eq. (5):

$$Y = 97.87 - 5.10x_1 + 0.67x_2 - 0.78x_3 - 11.40x_1x_2 - 6.90x_1x_3 - 1.07x_2x_3 - 14.33x_1^2 - 30.63x_2^2 - 33.70x_3^2 \quad (5)$$

where Y is the carbonation conversion of MSWI-BA; x_1 , x_2 , and x_3 correspond to independent variables of particle size (μm), L/S ratio (mL g⁻¹), and gas flow rate (L min⁻¹), respectively. Fig. 9(a) and (b) shows the relationships between the response and the operating variables were illustrated graphically by 2D-contour plots and 3D response surface, respectively. The maximum conversion with a MSWI-BA particle size of 125–350 μm was found to be 94.7% operated under an L/S ratio of 10 mL/g and a gas flow rate of 1.15 L/min at 25 °C for 120 min. It suggests that the developed surface model should be used to predict the conversion of MSWI-BA with reasonable well in the range of parameters investigated.

3.4. Estimation of energy consumption, and comparison of carbonation conversion

The energy consumption of the proposed process under optimized conditions was presented in Table 4. As shown in the Table, the total energy consumption for capturing 1 ton of CO₂ using the slurry reactor was estimated to be 180.3 kW h. Even if all the bottom ashes generated annually were utilized for

carbonation process, the total electricity required for operating the developed carbonation process would be as low as only 0.5% of total electricity produced using heat recovery from the Bali incinerator (i.e., 235 GW h as shown in Table 2).

In Table 5, the experimental results obtained in this study were compared with those reported in the literature. For instance, the aqueous carbonation of MSWI fly ash under a long reaction time (e.g., 72–240 h) exhibits an average weight gain of 7–12%, corresponding to a CO₂ capture capacity of 100–120 g per kg ash (Li et al., 2007; Wang et al., 2010). On the other hand, the aqueous carbonation process using MSWI-APC, (Cappai et al., 2012) achieved a CO₂ capacity of 200 g CO₂ per kg APC at a relatively mild operating condition (i.e., 20 °C for 3 h). The high performance was attributed to the active chemical properties of MSWI-APC. In general, the dry-carbonation process possessed relatively lower capture capacity of 86.7 g CO₂/kg MSWI-BA (corresponding to 24 L CO₂/kg MSWI-BA) than the aqueous-carbonation process (Rendek et al., 2006).

In this study, a higher carbonation conversion of MSWI-BA (i.e., 90.7%) can be achieved by aqueous carbonation with CRW at a relatively lower temperature (25 °C) and pressure (14.7 psia). Since grinding of MSWI-BA is an energy-intensive process, it would consume additional energy, generating emissions of CO₂. Therefore, CO₂ capture co-utilizing municipal solid waste incinerator bottom ash (MSWI-BA) and cold-rolling wastewater (CRW) should be critically evaluated by life cycle assessment for system optimization in our future research work. In addition, the effects of other contaminants in air emission from the MSWI also should be evaluated.

Table 5

Comparison of experimental results and operating conditions in the literature and those of this study.

Material type	Method	Properties of ash		Operating conditions						Performance		References
		CaO (%)	PSD (μm)	Liquid types	Pres. (bar)	CO ₂ concn. (%)	L/S ratio (mL/g)	Temp. (°C)	Time (min)	Carbonation conversion (%)	CO ₂ capture capacity (per kg waste)	
MSWI fly ash	Aqueous carbonation	36.3	– ^a	DI water	3	100	0.3	8–42	4320	35.1%	100 g CO ₂	Li et al. (2007)
MSWI fly ash	Aqueous carbonation	53.0	1–600	DI water	1	100	4	25	14,400	28.8%	120 g CO ₂	Wang et al. (2010)
MSWI APC (Sardinia, Italy)	Aqueous carbonation	308 ppm as Ca	– ^a	DI water	1	100	2.5	20	180	– ^a	200 g CO ₂	Cappai et al. (2012)
MSWI-BA	Dry carbonation moist. = 20%	16.3	4000	No liquid used	17	100	– ^a	25	180	67.7%	86.7 g CO ₂	Rendek et al. (2006)
MSWI-BA	Dry carbonation moist. = 65%	– ^a	710	No liquid used	3	100	0.3–0.4	25	1440	– ^a	31.9 g CO ₂	Fernandez Bertos et al. (2004a)
MSWI-BA	Aqueous carbonation	16.4	125–350	CRW	1	100	10	25	120	90.7%	102 g CO ₂	This study

^a Not specified.

4. Conclusions

In this study, the influences of different operating conditions including reaction time, CO₂ concentration, particle size, L/S ratio, and gas flow rate on carbonation of MSWI-BA were investigated. The carbonation rate decreased as reaction time increased. A highest conversion of 90.7%, corresponding to a capture capacity of 102 g CO₂ per kg of MSWI-BA, was achieved experimentally with a particle size of 125–350 μm and a reaction time of 120 min at 25 °C. Therefore, the annual CO₂ emission from the Bali incinerators could be expected to reduce by 6480 ton per year using the MSWI-BA carbonation process developed in this study. In addition, the observations of SEM, EDX and XRD show that the MSWI-BA was coated by crystallized calcite precipitation (i.e., CaCO₃) after the carbonation process. Based on the SCM results, the mechanism of carbonation reaction of MSWI-BA co-utilizing CRW in the slurry reactor should be ash-diffusion controlled, in which the diffusivity (D_e) ranges between 1.00×10^{-6} and 2.90×10^{-5} cm²/s.

Moreover, the effect of different operating parameters on carbonation conversion of MSWI-BA was statistically evaluated by RSM, where the predicted maximum conversion with a MSWI-BA particle size of 125–350 μm was 94.7% operated under an L/S ratio of 10 mL/g and a gas flow rate of 1.15 L/min at 25 °C and 120 min. Furthermore, the total energy consumption for capturing 1 ton of CO₂ using the slurry reactor was estimated to be 180 kW h, which was as low as only 0.5% of total electricity produced using heat recovery from the Bali incinerator. It was thus concluded that carbonation of MSWI-BA in a slurry reactor is a viable method for waste stabilization and CO₂ fixation due to its high carbonation conversion of MSWI-BA.

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