

산업폐기물의 가속 탄산화법을 이용한 CO₂ 고용화 및 중금속 안정화 특성 연구

Stabilization of Heavy Metal and CO₂ Sequestration in Industrial Solid Waste Incineration Ash by Accelerated Carbonation

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Abstract

In this study, an accelerated carbonation process was applied to stabilize hazardous heavy metals of industrial solid waste incineration (ISWI) bottom ash and fly ash, and to reduce CO₂ emissions. The most commonly used method to stabilize heavy metals is accelerated carbonation using a high water-to-solid ratio including oxidation and carbonation reactions as well as neutralization of the pH, dissolution, and precipitation and sorption. This process has been recognized as having a significant effect on the leaching of heavy metals in alkaline materials such as ISWI ash. The accelerated carbonation process with CO₂ absorption was investigated to confirm the leaching behavior of heavy metals contained in ISWI ash including fly and bottom ash. Only the temperature of the chamber at atmospheric pressure was varied and the CO₂ concentration was kept constant at 99% while the water-to-solid ratio (L/S) was set at 0.3 and 3.0 dm³/kg. In the result, the concentration of leached heavy metals and pH value decreased with increasing carbonation reaction time whereas the bottom ash showed no effect. The mechanism of heavy metal-

stabilization is supported by two findings during the carbonation reaction. First, the carbonation reaction is sufficient to decrease the pH and to form an insoluble heavy metal-material that contributes to a reduction of the leaching. Second, the adsorbent compound in the bottom ash controls the leaching of heavy metals; the calcite formed by the carbonation reaction has high affinity of heavy metals. In addition, approximately 5 kg/ton and 27 kg/ton CO₂ were sequestrated in ISWI bottom ash and fly ash after the carbonation reaction, respectively.

Key words: industrial solid waste incineration (ISWI) fly ash, bottom ash, accelerated carbonation, heavy metals, stabilization, CO₂ sequestration

● INTRODUCTION

With the development of industrial technology and improved living standards, the amount of waste being produced is also increasing and various hazardous substances pollute the environment (Jeon et al., 2009). Wastes are broadly categorized into domestic waste and industrial waste (industrial solid waste below), and they can also be categorized according to the source, emission characteristics, and hazardousness. Industrial solid waste is further categorized as general industrial waste, construction waste, and designated waste, and each type of waste is processed with a different method. Industrial wastes include sludge, incineration ash, waste oil, waste acid, waste alkali, waste rubber, waste synthetic resin, and other

wastes designated by the Ministry of Health and Welfare. Among various industrial wastes (waste acid, waste oil, waste synthetic resins, sludge, etc), sludge (dust, waste molding sand, ceramic parts) is incinerated at high temperature(above 980°C) to meet the toxicity destruction rate of 99.99%. However, most of the ash generated from the incineration is buried and this can also cause environmental problems (Ministry of Environment, 2012). In addition, a technology that stabilizes hazardous substances is required, because the incineration ash contains chlorine (Cl) and heavy metal components, similar to domestic waste incineration ash (Ahn et al., 2006; Van Gerven et al., 2005).

Stabilization of wastes through a carbonation reaction is currently being researched. The carbonation reaction is an exothermic reaction

that induces leaching of calcium ions from carbonation reaction materials as CO_2 is dissolved and precipitates carbonates, such as CaCO_3 (Lange et al., 1997). Such carbonation reaction is a continuous reaction that occurs through the following 8 stages (Maries et al.).

1. Diffusion of CO_2 into air
2. Penetration of CO_2 into solids
3. Dissolution of $\text{CO}_2(\text{g})$ into $\text{CO}_2(\text{aq})$
4. Conversion of $\text{CO}_2(\text{aq})$ into H_2CO_3 (rate-controlling step of carbonation reaction)
5. Ionization of H_2CO_3 into H^+ , HCO_3^- , CO_3^{2-}
6. Decomposition of solids (leaching of cations such as Ca^{2+})
7. Nucleation of carbonates
8. Precipitation of carbonates

The carbonation reaction is a diffusion controlling reaction in which carbon dioxide carbonates the surface of particle as it diffuses into a solid and expands its region. Carbonate precipitation on the outer surface and interior of the solid is a conventional reaction.

Stabilization of heavy metals is classified into weathering and accelerated carbonation. However, it is very difficult to obtain consistent carbonation conditions for the weathering method, where the reaction takes place with the CO_2 in the atmosphere, due to changes in temperature, relative humidity, and rainfall with respect to the change in seasons. Furthermore, the carbonation rate is very slow, and thus an accelerated carbonation

method, which utilizes a high concentration of CO_2 , is commonly used. The concentration of CO_2 , relative humidity, moisture content, temperature, and other factors are controlled in an accelerated carbonation process to yield the optimal carbonation reaction rate, and the CO_2 produced from the incinerator is retained in the wastes and it also offers the opportunity of CDM (Clean Development Mechanism) projects for CO_2 reduction in response to climate change (Nam et al., 2012; Lim et al., 2010). This study aims to stabilize industrial solid waste incineration bottom ash and fly ash containing high hazardous heavy metal content by employing accelerated carbonation, and measured the carbon dioxide sequestration of incineration ash caused by the carbonation reaction.

● EXPERIMENT METHOD

The incineration bottom ash and fly ash used in this experiment are generated from an incineration facility of Pyeongtaek-city in Gyeonggi, Korea. In order to obtain specimens that have not been through a weathering process, specimens that had been stored for approximately a day were used in the experiments. Moisture content of ISWI bottom ash and fly ash was measured according to the standard test methods. The masses of the specimens were measured prior to drying, and then measured again after 4 hours of drying

at 105°C. The moisture content was calculated through the mass differences of the specimens before and after drying. It was measured three times for each specimen and the average was taken. The moisture content of ISWI bottom ash and fly ash was 22.19% and 4.70%, on average, respectively.

The obtained specimens were dried at 105°C for 3 days in a large dryer and then sealed from the external environment. The step mentioned above was taken to minimize errors in the experiments possibly induced by weathering when the specimens are exposed to air. Incineration bottom ash of industrial solid wastes is composed of materials with various particle sizes. Therefore, the ash was separated through 6 standard sieves of +4.75, 2.36~4.75, 1.18~2.36, 0.6~1.18, 0.3~0.6, 0.15~0.3, -0.15 mm. Incineration fly ash is composed of fine particulates and it was separated using a 100 mesh.

Accelerated carbonation was conducted to stabilize the high content of hazardous heavy metals in industrial solid waste incineration ash, and carbonation reactions experiments were conducted using two methods with different solid-liquid ratio. Figure 1 schematically illustrates the equipment used in the experiments. The experiments were composed of dry carbonation, conducted with a solid-liquid ratio of 1:0.3 (Figure 1a), and wet carbonation, conducted with a solid-liquid ratio of 1:3 (Figure 1b); other conditions

of the experiments used here were room temperature, atmospheric pressure, carbon dioxide (purity .99%) injection at a rate of 1L/min, and stirring at 300rpm. The experiment of dry carbonation was carried out for 1 hour, and that of wet carbonation was terminated when the measured pH was below 7 and did not change any further.

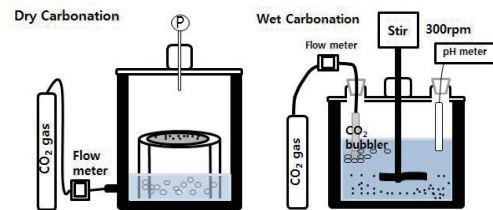


Fig. 1. Apparatus for accelerated carbonation.

distilled water of pH 5.8~6.3 was mixed with the specimen in a 1:10(W:V) ratio and the resultant solution was shaken for 5 hours under room temperature and atmospheric pressure with a shaker with approximately 200 shakes per minute and an amplitude of 4-5cm. It was then filtered through 1.0 μ m fiber glass filter paper, and the heavy metal concentration of the filtrate was measured with an Atomic Absorption Spectrophotometer (AAS, Shimadzu, AAS-60).

The ignition loss of the industrial solid waste incineration ash was analyzed with a Thermo Gravimetric/ Differential Thermal Analysis (TG/DTA, Shimadzu, DTG-60H) to measure the amount of carbon dioxide sequestration of industrial solid waste incineration ash. The TG/DTA analysis measures the change in mass of the solid specimens due to heat

over various ranges of temperatures. The change in mass is based on the principle that a state change in mass is due to the chemical components of a specimen decomposing into gaseous compounds and evaporating into the atmosphere. The mass loss can be calculated by measuring the mass of the specimen before and after the reaction. Table 1 illustrates the decomposition temperature of CaCO_3 , which is generally around $550\sim 800^\circ\text{C}$ (Lim et al., 2010).

Table 1. TGA decomposition of CaCO_3 (Lim et al., 2010)

	Ca(OH) ₂ TGA Decomposition	CaCO ₃ TGA Decomposition	Aragonite-calcite Conversion	Vaterite-calcite Conversion
Cole and Kroone (1960)		600-750 poorly crystallized 820 well crystallized		
Ramachandran et al. (1964)	464 (DTA)	850-950 calcite		
Taylor et al. (1985)	450-650			
Papadakis et al. (1991)	400-500	600-800		
Papadakis et al. (1992)	460			
Stem (2001) [62]		827-927 calcite	-460	350-400
Huijgen et al. (2005)-slag		>500		
Huntzinger (2006)	300-500	500-800		
Chang and Chen (2006)	425-550	550-950		

● RESULTS AND DISSUTIONS

Accelerated carbonation of industrial solid waste incineration ash

Table 2 shows the particle size distribution of

industrial solid waste incineration bottom ash, presenting a regular distribution ratio of each particle size.

Table 2. Particle size distribution of industrial solid waste incineration (ISWI) bottom ash (Unit : %)

Particle Size (mm)							
+4.75	2.36 ~4.75	1.18 ~2.36	0.6 ~1.18	0.3 ~0.6	0.15 ~0.3	-0.15	Total
11.5	9.7	12.1	15.6	21.6	12.6	16.9	100

Table 3 shows the particle size distribution of the incineration bottom ash and fly ash. Particles under 100 mesh particle size composed 99% of the incineration fly ash, and particles under 100 mesh particle size composed 17% of the incineration bottom ash. Heavy metals are generally contained in materials with a high content of small particle size, and calcium for the carbonation reaction is also included in materials with high content of small size particles. Therefore, specimens of incineration bottom ash and fly ash with particle sizes smaller than a 100 mesh were used for effective experiments.

Table 3. Particle size distribution of ISWI ash (Unit : %)

	Bottom Ash	Fly Ash
+100mesh(150μm)	83	1
-100mesh(150μm)	17	99

Table 4 shows the chemical compositions ISWI bottom ash and fly ash. The major components of ISWI bottom ash are 29.24% SiO_2 , 12.94% Al_2O_3 , 16.23% CaO , and 9.09% Fe_2O_3 . The major components of ISWI fly ash are 24.51% CaO , 16.2% Na_2O , 11.84% K_2O , and 21.22% SO_3 . X-ray fluorescence (XRF, Rigaku, Primus 2) and X-ray diffraction (XRD,

Rigaku, RU-200) were used in the analyses of the chemical compositions and crystalline structures of ISWI ash. Figure 2 illustrates the XRD crystalline analysis results. From the analyses the major crystalline structures of ISWI bottom ash are composed of SiO₂ (Quartz), CaAl₂Si₂O₈ (Anorthite), and CaCO₃ (Calcite), and the major crystalline structures of ISWI fly ash are composed of NaCl, Ca(OH)₂, CaSO₄, and NaOH.

Table 4. Chemical composition of ISWI ash (Unit: wt.%)

	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	MgO	Na ₂ O	K ₂ O	SO ₃	Loss on Ignition
Bottom Ash	29.24	12.94	16.23	9.09	2.56	1.29	1.94	5.67	12.77
Fly Ash	2.52	0.73	24.51	0.58	2.56	16.2	11.84	21.22	16.23

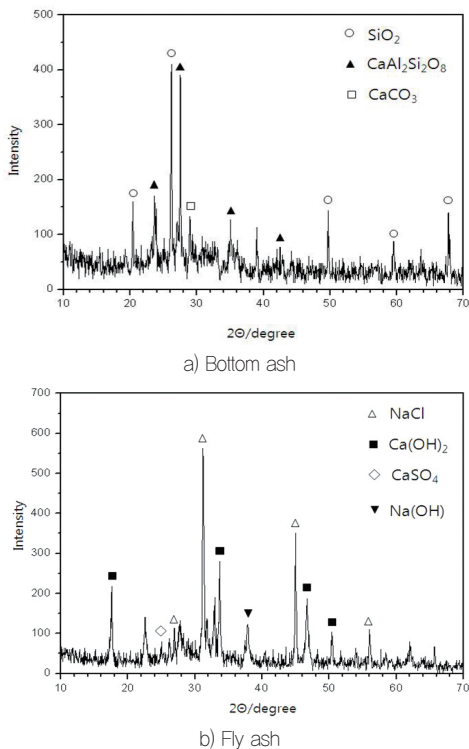


Fig. 2. XRD pattern of ISWI ash

Table 5 shows the concentration of leached heavy metals from ISWI bottom ash of each

particle size. The concentrations of heavy metals for each particle size were below the environment standard and Cr and Cd were not leached at all from the bottom ash.

Table 5. The concentration of leached heavy metals in ISWI bottom ash (Unit: mg/L)

Particle Size (mm)		Cr	Cu	Pb	Cd
		Total specimen	ND	0.2	0.3
Particle Size (mm)	+4.75mm	ND	0.2	0.3	ND
	2.36~4.75	ND	0.2	0.3	ND
	1.18~2.36	ND	0.2	0.3	ND
	0.6~1.18	ND	0.2	0.3	ND
	0.3~0.6	ND	0.2	0.3	ND
	0.15~0.3	ND	0.2	0.3	ND
	-0.15mm	ND	0.2	0.3	ND
Standard		1.5	3	3	0.3

※ ND = Not Detected

Table 6 shows the leaching concentration and total content of ISWI ash specimens with particle sizes smaller than a 100 mesh. ISWI bottom ash showed that the leached concentrations of metals were below the environment standard, but the fly ash showed very high Pb concentration well above the environment standard. Therefore, a stabilization process is required to prevent pollution caused by leaching of Pb, and an accelerated carbonation processing method was employed in the experiments for stabilization of ISWI bottom and fly ash.

Table 6. The concentration of leached (mg/L) and total (mg/kg) heavy metals in ISWI ash under 100mesh

	Cr		Cu		Pb		Cd	
	Leached	Total	Leached	Total	Leached	Total	Leached	Total
Bottom Ash (-100mesh)	ND	311	0.20	7773	0.30	6756	ND	25
Fly Ash (-100mesh)	ND	14.5	0.43	730.5	58.6	9195	0.04	239
Standard	1.5		3		3		0.3	

※ ND = Not Detected

Effect of carbonation reaction in ISWI ash

The pH values of ISWI bottom and fly ash were measured after carbonation reactions under wet carbonation conditions. Figure 3 shows the pH of ISWI bottom and fly ash before and after the carbonation reactions. The pH of the bottom and fly ash before the carbonation reactions was 10 ~ 12, but below 7 after the carbonation reactions. The decrease in pH is due to saturation of carbon dioxide by CO_3^{2-} ions, and this signals the termination of the carbonation reaction.

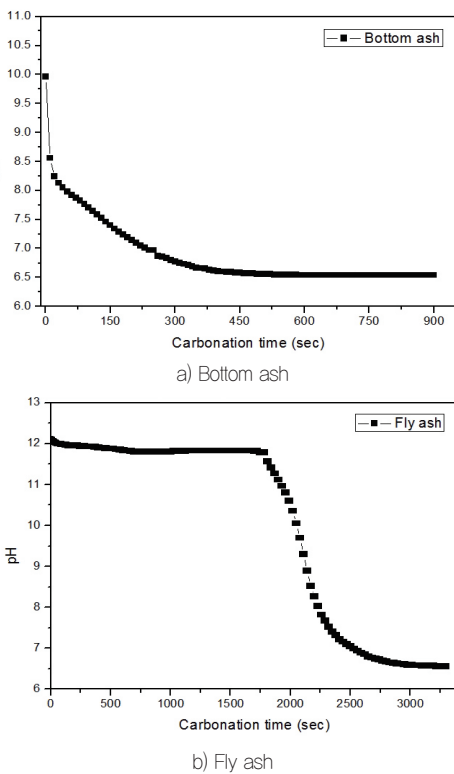


Fig. 3. Behavior of pH during accelerated carbonation

The changes in the mineral phases of the ISWI bottom and fly ash before and after the carbonation reactions were measured with

XRD and the results are illustrated in Figure 4. ISWI bottom and fly ash both showed increases in calcium carbonate(CaCO_3), and calcite, which did not exist in the initial state, was generated in fly ash. The calcite generation reflects the generation of Portlandite ($\text{Ca}(\text{OH})_2$), a hydration material, and carbon dioxide through the carbonation reaction. In addition, the intensity of NaCl peak was reduced after the carbonation reaction.

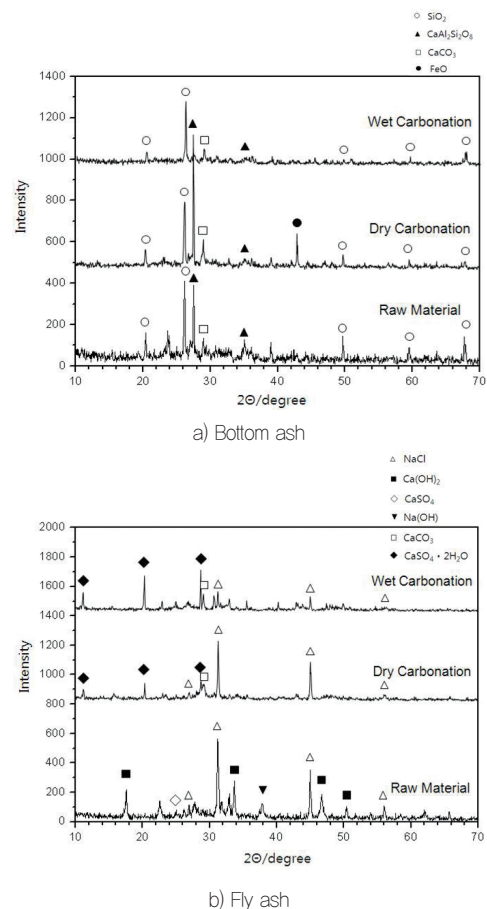


Fig. 4. XRD pattern of carbonated ISWI ash

Table 7 shows the leaching content of the heavy metals from ISWI bottom and fly ash

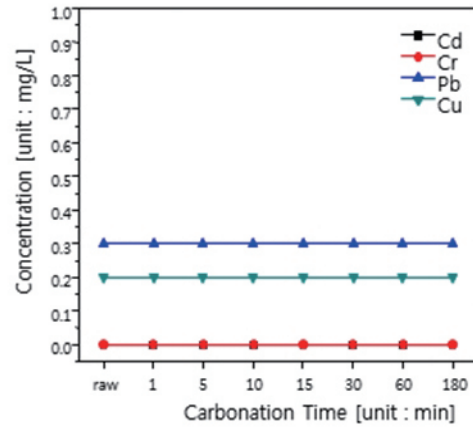
smaller than a 100 mesh after carbonation reactions through accelerated carbonation. Although the ISWI bottom ash did not show a substantial difference, the very high Pb leaching in fly ash, 58.6mg/L, was reduced to 0.18 and 0.20 after wet and dry carbonation, respectively, and these values are below the environment standard. Therefore, Pb, a hazardous heavy metal, is determined to be stabilized by wet and dry carbonation reactions and ISWI ash can thus be available for recycling.

Table 7. Leaching concentration of heavy metals in ISWI ash with accelerated carbonation (Unit : mg/L)

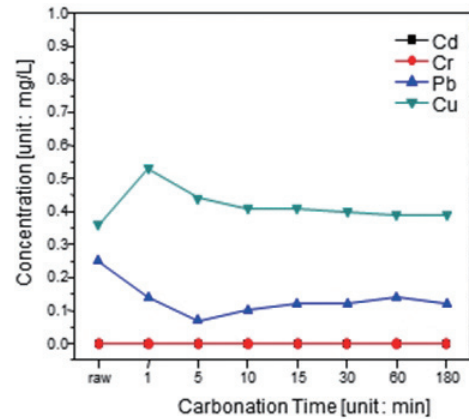
		Cr	Cu	Pb	Cd
Bottom Ash	Raw	ND	0.2	0.3	ND
	Dry Carbonation	ND	0.2	0.2	ND
	Wet Carbonation	ND	0.2	0.2	ND
Fly Ash	Raw	ND	0.43	58.6	0.04
	Dry Carbonation	ND	0.45	0.18	0.03
	Wet Carbonation	ND	0.2	0.2	ND
Standard		1.5	3	3	0.3

※ ND = Not Detected

Figure 5 illustrates Cr, Cu, Pb, and Cd leaching concentrations of bottom ash with respect to the dry and wet carbonation times. The concentrations of each heavy metal were lower than the international environment standard and did not show any significant change in leaching. However, the leaching of Pb stabilized as time elapsed. These results appear to be due to difficulty in measuring the carbonation effect at very low initial heavy metal concentrations.



a) dry and

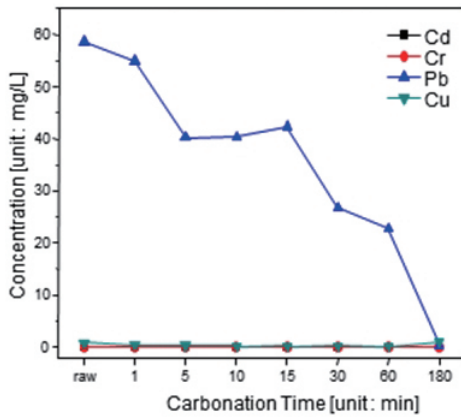


b) wet carbonation.

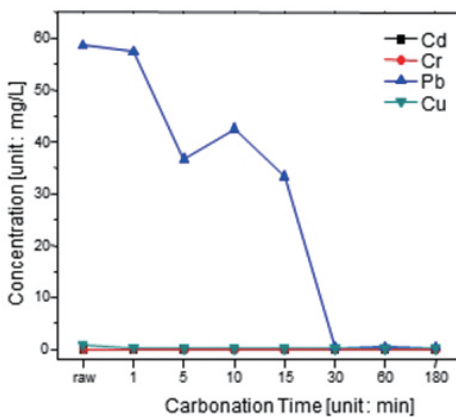
Fig. 5. Leaching behavior of heavy metals in ISWI bottom ash vs. reaction time using

Figure 6 illustrates the Cr, Cu, Pb, and Cd leaching during dry and wet carbonation. The behavior of Cu during dry carbonation reflected a slow carbonation effect taking place at an initial concentration of 0.85mg/L, and the leaching decreased. However, the concentration increased to 0.93mg/L and the leaching showed an increasing trend when the reaction time was increased to 180 minutes. This reveals that carbonation time is important for the stabilization of Cu of ISWI fly ash.

In addition, Pb yielded the best result in the stabilization of fly ash heavy metals through carbonation, showing leaching of 0.36mg/L, which is significantly lower than the international environment standard of 3mg/L.



a) dry and



b) wet carbonation

Fig. 6. Leaching behavior of heavy metals in ISWI fly ash vs. reaction time using

The behavior of the heavy metals in wet carbonation showed an excellent carbonation effect in Cu from the initial phase as leaching decreased from 0.82mg/L. The leaching was almost identical from the reaction time of 5 minutes to 180 minutes. Therefore, Cu

carbonation must have been completed by around 5 minutes of reaction, and prolonged reaction appears to be unnecessary. In addition, as in the dry carbonation results, Pb of the fly ash yielded a significant stabilization effect. After carbonation of 180 minutes, the Pb concentration dropped from an initial value of 168.85mg/L to very low leaching of 0.34mg/L, which is significantly lower than 3.0mg/L.

Behavior of chlorine in ISWI during washing process

As Figure 4 illustrates, NaCl did not exist in the bottom ash but an abundant amount was present in the fly ash. This difference in the presence of chlorine arises from the mixing of domestic waste and industrial solid waste during the collection process. Therefore, this study on the behavior of chlorine targets fly ash. As figure 7 illustrates, the chlorine analysis of ISWI fly ash after washing shows that most of the NaCl in ISWI fly ash is leached. In addition, with an abundant supply of water, it was verified that most of the chlorine could be eliminated with just one washing.

As the results showed above, most of the chlorine could be eliminated through washing treatment, and this study verified the leaching of dissolved chlorine with respect to time for fly ash. As Figure 8 illustrates, the analysis of residual chlorine shows that most of the chlorine was leached after 1 minute after the reaction, and only 222mg/L of chlorine was

present in the fly ash after 60 minutes.

Therefore, with the results from Figure 7 and Figure 8, it was verified that wet carbonation of ISWI fly ash (under conditions identical to those of the washing experiment) not only stabilizes the heavy metals but also eliminates NaCl after the carbonation.

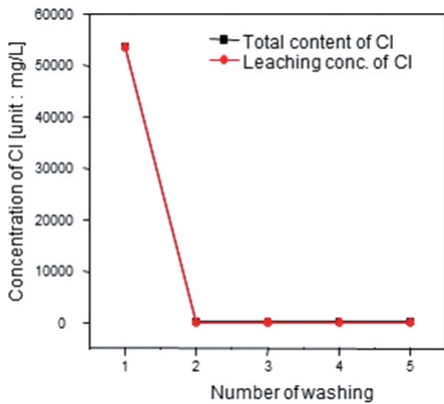


Fig. 7. Total content and leaching concentration of Cl in ISWI fly ash after washing process according to the number of washings

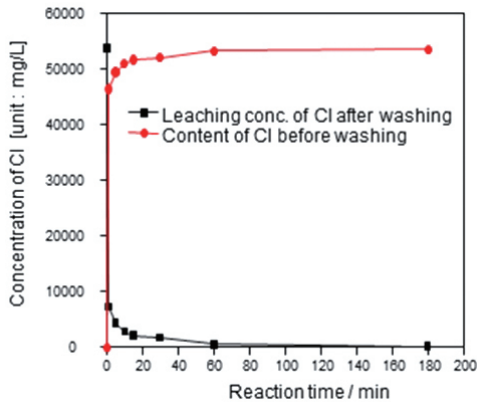


Fig. 8. Content and leaching concentration of Cl in ISWI fly ash after washing process versus the reaction time

CO₂ sequestration in ISWI ash after carbonation reaction

CO₂ sequestration per 1kg based on the

carbonation rates revealed that the CO₂ sequestration of ISWI bottom ash was approximately 3.1g-CO₂/kg and that of the fly ash was approximately 19.2g-CO₂/kg, which is 6 times higher than that of the bottom ash. In addition, the bottom and fly ash processed with wet carbonation both yielded about twofold higher CO₂ sequestration than that processed with dry carbonation (Table 8).

Table 8. Carbonation rate and CO₂ sequestration in ISWI ash

	Carbonation	CO ₂ Sequestration (kg-CO ₂ /ton-waste)
Bottom Ash	Dry Carbonation	3.1
	Wet Carbonation	6.8
Fly Ash	Dry Carbonation	19.2
	Wet Carbonation	34.6

Most of the carbonation reaction was completed when the reaction time passed 60 minutes, and CO₂ sequestration after 60 minutes did not show a significant change with the carbonation reaction of ISWI ash smaller than a 100 mesh. In addition, the wet carbonation CO₂ sequestration (dry: 3.2g-CO₂/kg, wet: 7.0g-CO₂/kg-reaction time: 60minutes) was approximately twice as large as that of dry carbonation (Figure 9, a). Like bottom ash, the carbonation of the fly ash was almost completed when the carbonation reaction time passed 60 minutes, and no change in CO₂ sequestration occurred after 60 minutes. In addition, CO₂ sequestration for 1kg of ISWI bottom ash was 19.7g-CO₂/kg from dry carbonation and 35.0g-CO₂/kg from wet carbonation. The sequestration from wet

carbonation was clearly higher than that of dry carbonation (Figure 9, b).

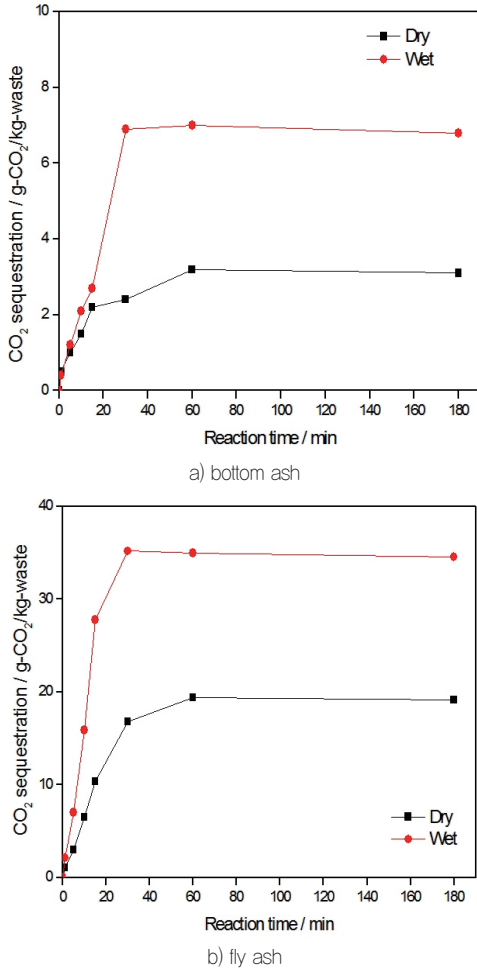


Fig. 9. CO₂ sequestration rate vs. carbonation time in ISWI

CONCLUSION

Industrial solid waste incineration (ISWI) bottom ash and fly ash contain large amounts of heavy metals and chlorine, and leaching of heavy metals of ISWI ash has become an issue due to environmental and recycling concerns. Therefore, the stabilization of heavy metals,

chlorine, and expansion substances has been conducted in this study to resolve these issues. The major components of ISWI bottom ash were 29.24% SiO₂, 12.94% Al₂O₃, 16.23% CaO, and 9.09% Fe₂O₃, and the major components of the ISWI fly ash were 24.51% CaO, 16.2% Na₂O, 11.84% K₂O, and 21.22% SO₃. In addition, the major crystalline structures of the bottom ash were SiO₂ (Quartz), CaAl₂Si₂O₈ (Anorthite), and CaCO₃ (Calcite), and the major crystalline structures of the fly ash were NaCl, Ca(OH)₂, CaSO₄, and NaOH. Heavy metal leaching from the bottom ash was below the environment standard, but the fly ash yielded a significantly high concentration of leached Pb of 58.6mg/L, which is well above the environment standard. Strong alkaline ISWI ash was neutralized, and the high Pb leaching from the fly ash was stabilized to below the environment standard through dry and wet accelerated carbonation. In addition, portlandite, an expansion substance, and NaCl, salt, were also stabilized. The carbonation rates of ISWI bottom ash and fly ash by accelerated carbonation yielded a fly ash carbonation rate that was approximately 4.6 times higher than that of the bottom ash. This difference in the carbonation rates appears to arise from the abundant hydration materials such as Ca(OH)₂ in the ISWI fly ash. CO₂ sequestration of the bottom ash and the fly ash based on the carbonation rates yielded maximum values of 6.8kg-CO₂/ton-waste and 34.6kg-CO₂/ton-waste for the bottom ash

and fly ash, respectively. The fly ash yielded approximately 5 times higher sequestration than that of the bottom ash.

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