

Study on the Recycling Technology of Inorganic Wastes Generated from Industry Fields by CO_2 Gas

Summary

In this research, we focused on the aspect that bottom ashes are materialized and utilized to recycle with using CO_2 gas. Bottom ashes which are composed of daily wastes contain lots of chlorides and heavy metals. Therefore, technology of eliminating chlorides and stabilizing heavy metals is necessary. We used CO_2 gas, which causes greenhouse effect, to eliminate heavy metals and chloride. After carbonating process, we applied heat to treated bottom ashes with $1350^\circ C$ to synthesize mineral of Calcium-Aluminium system. The mineral is known for its special ability that it can be converted into Ettringite. Ettringite is widely known as a substance which can solidify heavy metals. In this study, we synthesized Ettringite and examined mechanisms of behavior of heavy metals when they are solidified. Especially, we tried to examine mechanisms of Zinc and Cadmium, which is supposed to be substituted on calcium ions' sites, and Copper and Iron, which is supposed to be substituted on Aluminium ion's sites. We could use ultrasonicator and magnetic stirrer to inject energy while synthesizing Ettringite in order to increase reaction's rates. However, when we use ultrasonicator, we found that it injects too much energy. Therefore, it breaks the crystal of Ettringite. Also, we were able to discover that Zn, Cu, and Fe ions are substituted for ions in Ettringite, not just attached to the surface of Ettringite. However, we could find that when we injected Cadmium, a lot of new, unreacted gypsum was produced. From this result, we were able to find that Cadmium controls the growth of crystal.

1. Introduction

Daily wastes are treated into three ways - reclamation, incineration, and recycling. The ratio of reclamation has decreased while the ratio of incineration has continuously increased because of many policies such as 'Establishment of Incinerators' and 'Comprehensive Program of Administration of National Wastes'. Therefore, the amount of cinders, especially that of bottom ashes has increased. The total amount of cinders was about 400,000 tons, composed of 350,000 tons of bottom ashes and 50,000 tons of fly ashes in 2004. However, technology of treating bottom ash is not enough to manage all bottom ashes.

Bottom ashes are usually classified into domestic wastes. Therefore, they are reclaimed, but many countries such as Europe and United States use them as an alternative aggregate after treating them

with proper methods. Especially, France has encouraged recycling bottom ashes because France doesn't have enough land. So, 40 percent of bottom ashes is used to build roads. However, in Korea, only 3~4 percent of bottom ashes is used to make bricks, while the rest is reclaimed.

Lots of substances exist in bottom ashes since they are remnants of daily wastes. Chlorides are also compounds in bottom ashes, and it causes a lot of problems like rusting ionic equipments and causing soil pollution when it dissolved in rain. Also, when they dissolve in water, the pH value of the solution becomes 9.5~12, which is easy value for the solution to dissolve heavy metals in rocks and soil. Also, the alkali solution tends to cause expansion of concrete by reacting on silica to produce alkali-silica gel. The expansion causes cracks of concrete structures.

To solve these problems, carbonizing methods can be used. Reacting bottom ashes on carbon dioxide, we can reduce the amount of carbon dioxide in air. Furthermore, we can stabilize heavy metals in bottom ashes, neutralize the pH value of the solution, and synthesize minerals of Calcium-Aluminate system. The mineral can be converted into Ettringite, which is effective to solidify heavy metals by hydrating.

Now, the importance and necessity of Ettringite will be mentioned.

Heavy metals are defined as metals which specific gravity is larger than 5.0. For instance, Cobalt, Copper, Iron, and Zincs are very important metals for organisms to maintain functions. However, in general, most heavy metals are fatal such as lead, mercury, and cadmium. Although only 0.1 percent of crust is composed of heavy metals, lots of damages by heavy metals are reported in many areas. So, interests of world-wide nations have increased and many policies have been made. For example, EPA considered the technology of solidification and stabilization of heavy metals as the most important technology.

The following explanation will show the reason for heavy metal pollution. First of all, factory wastes contain a lot of heavy metals. There are various kinds of waste water. So, it is a very crucial problem. Especially, waste water from electronic, fertilizer, toy, battery, and car factory is considered as a representative reason for the pollution.

Every day, lots of metals are obtained, too. Typical phenomenon is that the density of lead in stream increases after raining since dusts and leads are flowed into stream by rainwater. Also, chemical fertilizer contains potassium, magnesium, silicon, and manganese. So, if we use large amounts of fertilizers, they can toxicate soil and underground water which can cause fatal problems. Especially, if manganese is stored in brain and liver, it can cause decline abilities of production and growth. In addition, acidification of rainwater caused increments of dissolving heavy metals.

Lastly, water from dead pit causes lots of problems. It causes not only heavy metal pollution but also water pollution. Thus, it taints soil, inhabitation and ocean. It also has an effect on fishes, insects and

higher lives. For example, Itai-itai disease was appeared because of waste water from nearly Zinc mines.

Hence, development of technologies to solidify heavy metals is acutely needed. Especially pollutants are appeared in many different ways, so there is a limitation. In this study, we eliminated heavy metals and chlorides in bottom ashes with using carbon dioxide, one of gases which cause greenhouse effects. After that, we synthesized Ettringite which is known for ability to stabilize and solidify heavy metals with hydration reaction. Also, examining mechanism of solidifying heavy metals during the reaction of Ettringite is purpose of our study. Especially, Zinc, Cadmium, Copper and Iron are used to solidify. Experiences were proceeded in KIGAM with Dr. Ahn, Dr. Cho, and DR. Yoo.

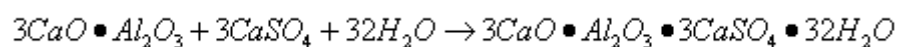
2. Theory

A. Ettringite

Ettringite, which is formed by hydrating $\text{Ca}_3\text{Al}_2\text{O}_6$ with gypsum, behaves as a host for metallic pollutants to immobilize waste ions through sorption, phase mixing and substitution. Therefore, Ettringite is one of useful materials for waste treatments.

1) Formation

Ettringite is formed within a few minutes of the start of cement hydration. The aluminate phase reacts with water in the presence of calcium hydroxide to form Ettringite. The reaction equation is as follows.



According to certain references, if we synthesize Ettringite with ultrasonicator or magnetic stirrer, then we can increase surface of reactants to contact and react with others. Also, we can increase collide frequency. Thus, if we synthesize Ettringite with ultrasonicator or magnetic stirrer, then the rate of reaction would be increased.

2) Structure

Ettringite forms hexagonal prismatic crystals. These crystals have two distinct structural components. One is

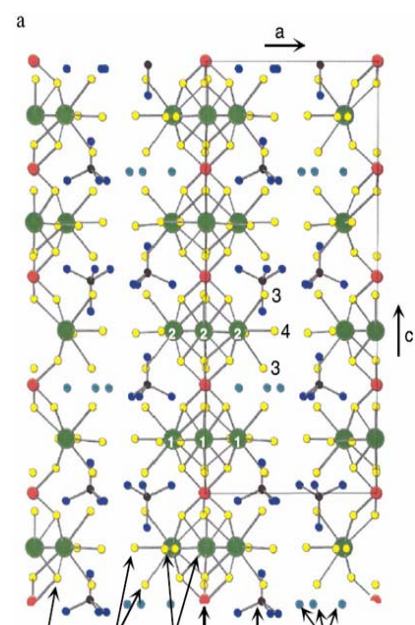


Figure 1 The structure of Ettringite

columns that have chemical formula, ' $[\text{Ca}_6[\text{Al}(\text{OH})_6]_2 \cdot 24\text{H}_2\text{O}]^{6+}$ '. The other is channels that have chemical formula ' $[(\text{SO}_4)_2 \cdot 2\text{H}_2\text{O}]^{6-}$ '. More circumstantially, the column consists of octahedral $\text{Al}(\text{OH})_6$ alternating with triangular groups of edge-sharing CaO_6 polyhedra. In addition, the channel contains four sites per unit of the column structure which contains six calcium atoms. Three of these sites are occupied by SO_4^{2-} and one by two H_2O molecules.

3) Waste Ion Immobilization

Ettringite, which has chemical formula $\{[\text{Ca}_6[\text{Al}(\text{OH})_6]_2 \cdot 24\text{H}_2\text{O}]^{6+}\}$, can withstand deviations in composition without a change in structure. This compositional change occurs on the crystal chemical level in the form of ionic substitution. All crystal chemical substitutions in minerals occur at aluminum and sulfate sites. However, according to a number of reports, there is a possibility that calcium-site and hydroxyl-site occur. Site limitations for immobilization are as follows.

Table 1 Classification of ions on each site in Ettringite which can be substituted.

(Ca^{2+}) site	(Al^{3+}) site	(SO_4^{2-}) site	(OH^-) site
Sr^{2+}	Cr^{3+}	$\text{B}(\text{OH})_4^-$	O^{2-}
Ba^{2+}	Sr^{4+}	CO_3^{2-}	
Pb^{2+}	Fe^{3+}	Cl^-	
Cd^{2+}	Mn^{3+}	OH^-	
Cs^{2+}	Ni^{3+}	CrO_4^{2-}	
Ni^{2+}	Co^{3+}	AsO_4^{3-}	
Zn^{2+}	Ti^{3+}	SeO_4^{2-}	
	Cu^{3+}	VO_4^{3-}	
		BrO_3^-	
		NO_3^-	
		MoO_4^{2-}	
		ClO_3^-	
		SO_3^{2-}	
		IO_3^-	

Moreover, there are also size limitations for each site which can be occurred substitutions of ions in Ettringite. According to Shannon and Prewitt ionic radius, size ranges for each site are as follows.

Table 2 Size limitations on each site in Ettringite.

Site	Size limitations
Ca^{2+}	0.90 Å to 1.42 Å
Al^{3+}	0.42 Å to 0.76 Å

SO_4^{2-}	0.62 Å to 3.71 Å
OH^-	0.62 Å to 1.38 Å

3. Experimental Method

A. Pre-treatment and recycling for bottom ash with CO_2 gas

In order to make Calcium-Aluminate minerals, we should treat bottom ashes with pre-treatment process because bottom ashes have two problems. One is that pH value of bottom ashes ranges between 11.9 and 12.4. In addition, the other is that because heavy metals in bottom ashes are unstable, secondary environmental pollution is derived via eluted heavy metals. So, in order to recycle bottom ashes, we should treat them with pre-treatment process.

We used bottom ashes at DaDePo, Haeundae-gu, Busan, Korea. The chemical composition is as follows.

Table 3. Chemical composition of bottom ashes at DaDePo, Haeundae-gu, Busan, Korea

Components	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	MgO	CaO	Na_2O	K_2O	MnO	P_2O_5	LOI	Cl
Ratio(%)	7.29	9.33	0.74	1.28	1.37	36.07	2.68	0.59	0.19	3.10	28.06	3.8

1) Stabilization of chromium in bottom ashes by CO_2 gas

We dissolved 40 grams of bottom ashes by magnetic bars in 400 ml of water at normal temperature. Then, we injected CO_2 with flow rate at 1.0L/min in order to occur carbonate reaction. After 2 hours, 4 hours, and 24 hours later, we took out 5 grams of bottom ashes respectively. Then, we measured the amount of chromium in each sample by using the equipment 'ICP-AES'. Finally, we analyze the amount of chromium in bottom ashes through time.

2) Removal of chlorides in bottom ashes by CO_2 gas

Like the first experiment, we dissolved 40 grams of bottom ashes by magnetic bars in 400ml of water at normal temperature. Then, we injected CO_2 with flow rate at 1.0L/min in order to occur carbonate reaction. After 15 minutes, 30 minutes, and 120 minutes later, we took out 5 grams of bottom ashes respectively. Then, we measured the amount of chlorides in each sample by using the equipment 'XRD'. Finally, we analyze the efficiency of removing the chlorides in bottom ashes through time.

3) Synthesizing Calcium-Aluminate minerals from bottom ashes treated with pre-treatment process.

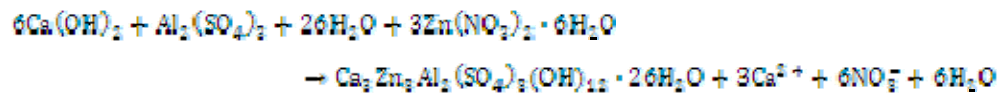
Calcium-aluminate minerals have the chemical composition which the ratio between moles of calcium and moles of aluminium is 3 : 1. However, our target materials, bottom ashes from DaDePo, Haeundae-gu, Busan, Korea have the chemical composition that the ratio between moles of calcium and moles of aluminium is 2.8 : 1. So, we added sludges which have some amounts of aluminium into 20.2 grams of bottom ashes in order to make the chemical composition which the ratio between moles of calcium and moles of aluminum is 3:1. Then, we heated it 1 hour at 800°C. Moreover, after that, we also heated it 2 hours at 1350°C. Finally, we hydrated it with lots of water. Eventually, we analyzed the chemical composition by using the equipment 'XRD' whether Ettringite is formed or not.

B. Synthesizing Ettringite and immobilization of heavy metals.

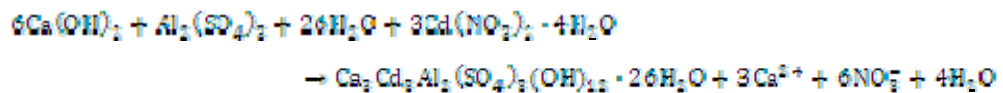
1) Solidification of heavy metals with using reaction of Ettringite I.

We tried to solidify heavy metals while synthesizing Ettringite. Therefore, we used ultrasonicator and magnetic stirrer to perform the reaction effectively, and compared with each other.

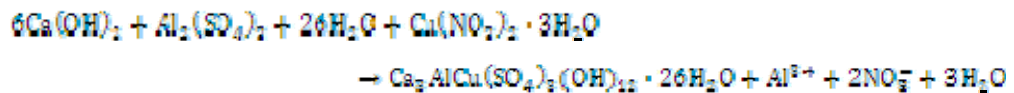
A) Solidification of Zinc in the Ettringite's structure with using ultrasonicator



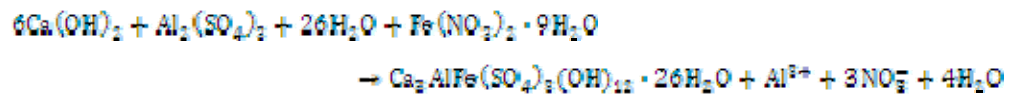
B) Solidification of Cadmium in the Ettringite's structure with using ultrasonicator.



C) Solidification of Copper(II) in the Ettringite's structure with using ultrasonicator.



D) Solidification of Iron(III) in the Ettringite's structure with using ultrasonicator.



In the experiment A) and B), zinc and cadmium are substituted on calcium ion's site. We injected only half of zinc and cadmium ions than that of calcium during the reaction to understand how much ions can be solidified in the Ettringite's structure.

In the experiment C) and D), copper(II) and iron(III) are substituted on aluminium ion's site. We injected only half of copper(II) and iron(III) ions than that of aluminium during the reaction to understand how much ions can be solidified in the Ettringite's structure.

In experiment A), B), C), and D), we filtered produced solutions with buncher funnel and got crystals of Ettringite which solidified four different kinds of heavy metals. We analyzed the chemical composition of the crystal with the equipment 'XRD' and the chemical composition of the solution which is filtered by funnel with the equipment 'ICP'.

2) Solidification of heavy metals with using reaction of Ettringite II.

- A) Solidification of Zinc in the Ettringite's structure with using magnetic stirrer.
- B) Solidification of Cadmium in the Ettringite's structure with using magnetic stirrer.
- C) Solidification of Copper(II) in the Ettringite's structure with using magnetic stirrer.
- D) Solidification of Iron(III) in the Ettringite's structure with using magnetic stirrer.

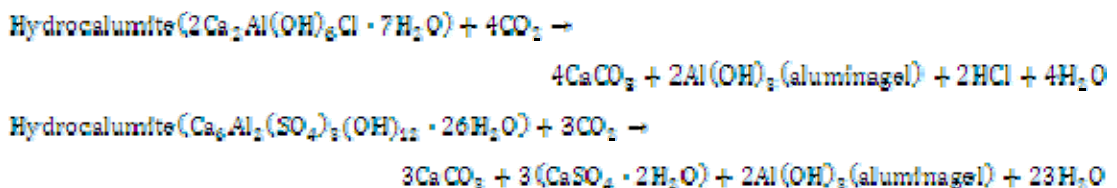
In the experiment A), B), C), and D), we did the same experiment of experiment 1) with using magnetic stirrer instead of ultrasonicator. And we filtered produced solutions with buncher funnel and got crystals of Ettringite which solidified four different kinds of heavy metals. We analyzed the chemical composition of the crystal with the equipment 'XRD' and the chemical composition of the solution which is filtered by funnel with the equipment 'ICP'.

4. Results and analysis

A. Pre-treatment and recycling for bottom ashes with CO_2 gas

1) Stabilization of chromium in bottom ashes by CO_2 gas

According to below graph, the amount of chromium in bottom ashes decreased during early 10 minutes. However, it increased after 10 minutes. Because lots of chromium are adhered by one of the major materials in bottom ashes, Ettringite and Hydrocalumite, it was dissolved in water for early 10 minutes. However, after 10 minutes, since carbonate reaction decomposed Ettringite and Hydrocalumite as follows, a lot of chromium is adhered by alumina gel.



Therefore, a lot of chromium is stabilized and immobilized by carbonate reaction.

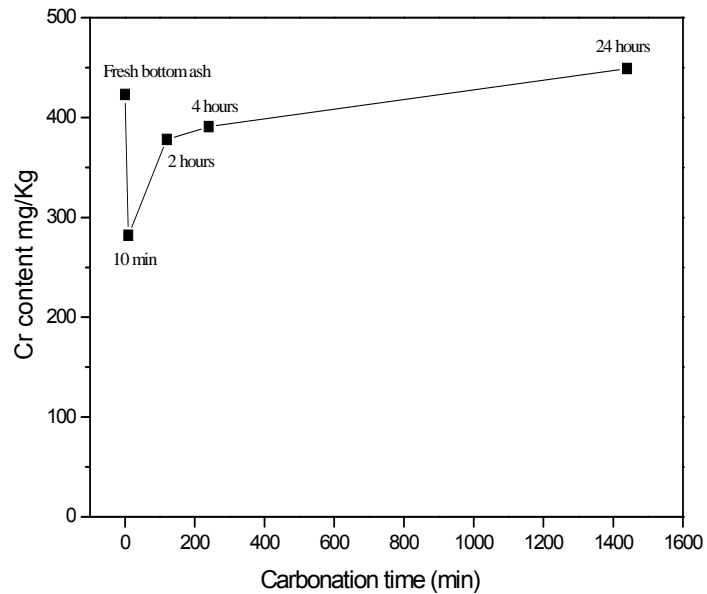
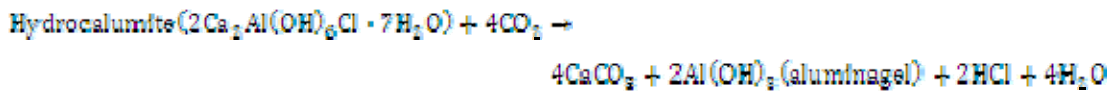


Figure 2 The graph of the movement of chromium in bottom ashes during carbonate reaction

2) Removal of chlorides in bottom ashes by CO_2 gas

According to analysis with the equipment 'XRD', the amount of Hydrocalumite ($2\text{Ca}_2\text{Al}(\text{OH})_6\text{Cl} \cdot 7\text{H}_2\text{O}$) in bottom ashes decreased quickly. However, the amount of calcite() increased quickly. Especially, after 120 minutes, the amount of hydrocalumite was completely disappeared. It means that chlorides in bottom ashes are completely removed because most chlorides exist in hydrocalumite and they are decomposed by carbonate reaction as follows.



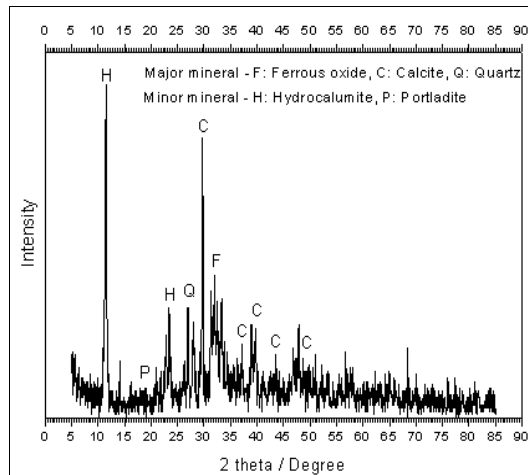


Figure 3.1. XRD patterns of Bottom ashes that doesn't have carbonate reaction.

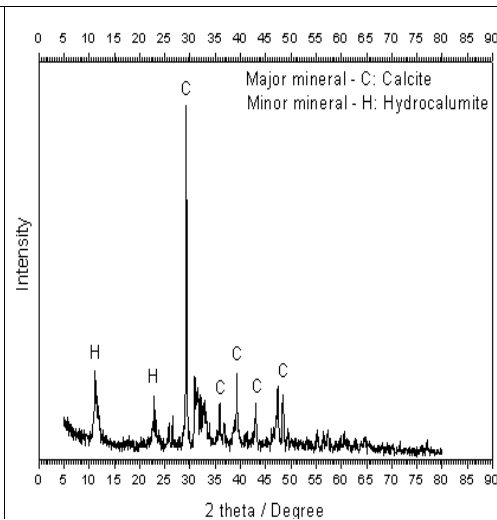


Figure 4.2. XRD patterns of Bottom ashes that have carbonate reaction during 15 minutes.

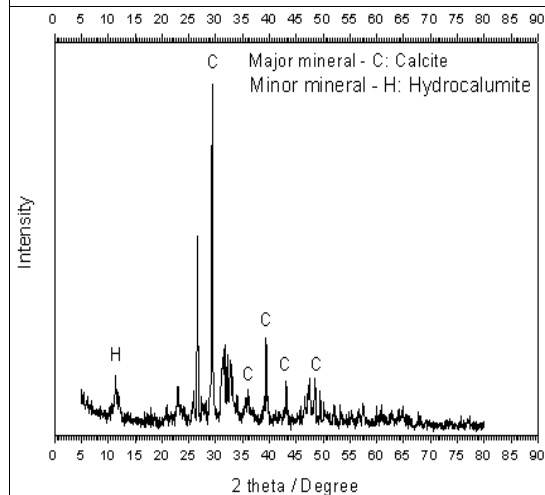


Figure 5.3. XRD patterns of Bottom ashes that have carbonate reaction during 30 minutes.

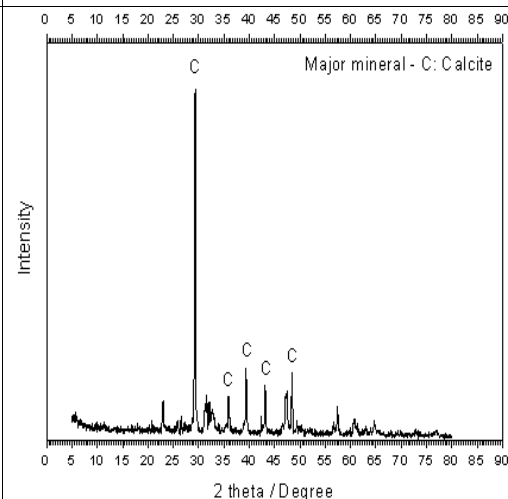


Figure 6.4. XRD patterns of Bottom ashes that have carbonate reaction during 120 minutes.

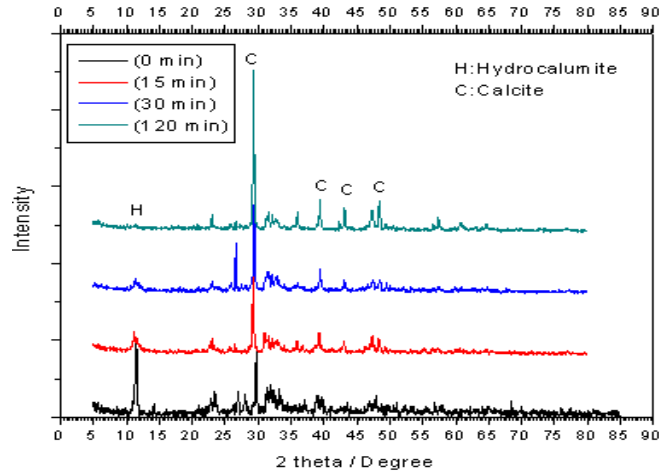
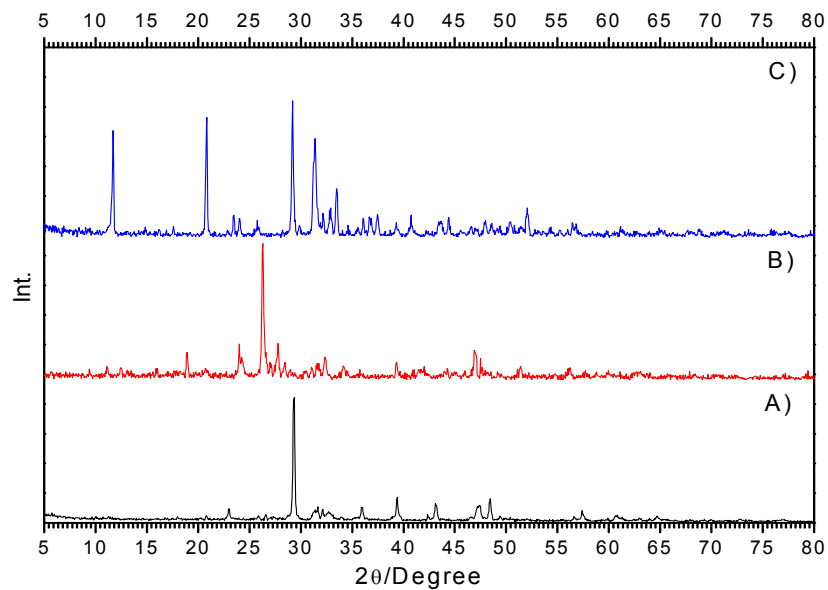
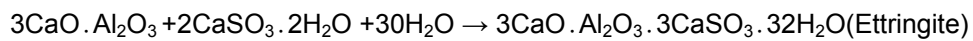


Figure 7.5. XRD patterns of bottom ashes through carbonate reaction's time.

3) Synthesizing Calcium-aluminate minerals from the bottom ash treated with pre-treatment process.

In this experiment, we synthesize calcium-aluminate minerals that can be converted into Ettringite. We use the bottom ash and water as reactants which is pre-treated by previous 2 experiments. Below XRD patterns show our experimental data. According to analysis for graph C, finally we made Ettringite.



**Figure 4. A) Bottom ashes treated with pre-treatment process.
B) Bottom ashes treated with pre-treatment process and heating process
C) Bottom ashes treated with pre-treatment process, heating process, and hydrating process.**

B. Synthesizing Ettringite and solidification of heavy metals.

1) Confirmation of solidification in Ettringite with using ICP.

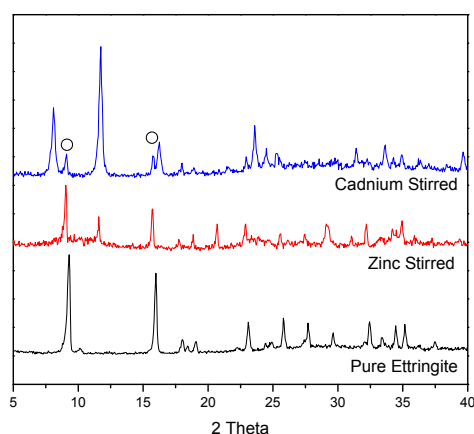
Heavy metal	Cd	Zn	Cu	Fe
the amount of substances in solution	486	920	570	317
the amount of substances after filtering (ultrasonication)	<0.01	0.02	<0.01	<0.01
the amount of substances after filtering (stirring)	0.07	0.05	<0.01	<0.01

The upper table shows the result of the amount of heavy metals in solutions. Solutions treated with both ultrasonication method and magnetic stirring method had few heavy metals. Therefore, we could make two guesses. One is that heavy metals were just attached on the surface of Ettringite. The other is that heavy metals were substituted on calcium's sites and aluminium ion's sites. However, we washed the crystal with distilled water much time. Therefore, it is impossible to attach on the surface. Therefore, we can conclude that heavy metals were immobilized into the Ettringite's structure. In other words, Ettringite can stabilize heavy metals.

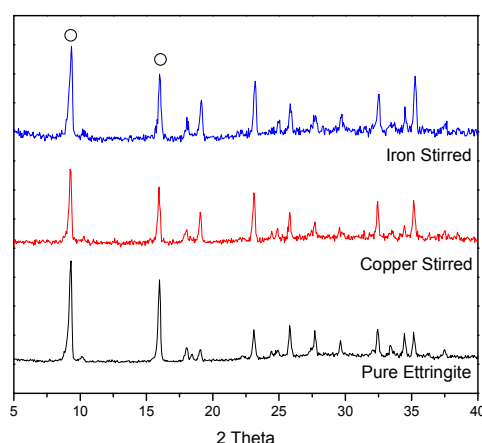
However, less amount of heavy metal ions were detected when we used ultrasonication method rather than magnetic stirring method. Hence, when we solidify heavy metals during synthesizing Ettringite, it is more efficient to use sonicator.

2) Comparing the shape of XRD peaks with crystals of each other methods.

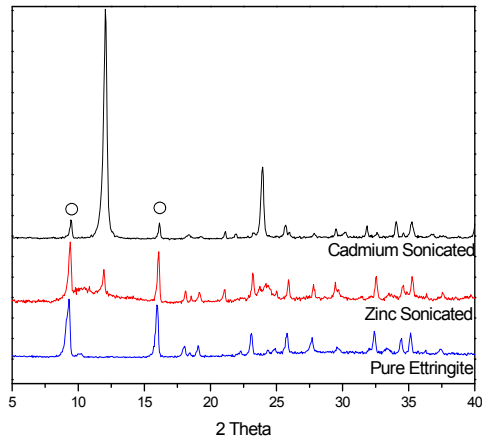
A.



B.



C.



D.

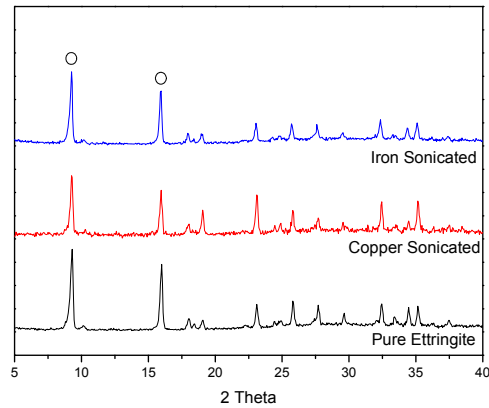


Figure 5. XRD Patterns of Ettringite which immobilized heavy metals

Table 5. Crystalline characteristic of Ettringites which solidified four kinds of heavy metals.

		Strongest Peak		2nd Strongest Peak		3rd Strongest Peak	
		2 θ /°	d[100], Å	2 θ /°	d[110], Å	2 θ /°	d[114], Å
Soni-cated	Pure	9.3	9.501	15.95	5.552	32.4	2.761
	Zinc	9.4	9.400	16.1	5.500	23.2	3.831
	Copper	9.1	9.710	15.8	5.604	22.9	3.880
	Iron	9.25	9.552	15.95	5.552	32.35	2.765
Stirred	Pure	9.3	9.501	16	5.534	25.8	3.450
	Zinc	9.25	9.552	15.95	5.552	23.1	3.847
	Copper	9.25	9.552	15.95	5.552	23.1	3.847
	Iron	9.35	9.450	16	5.534	23.2	3.831

Table 6. The change of lattice distance as heavy metals immobilized.

		Strongest Peak		2nd Strongest Peak		3rd Strongest Peak	
		2 θ /°	d[100], Å	2 θ /°	d[110], Å	2 θ /°	d[114], Å
Soni-cated	Zinc - Pure	0.1	-0.101	0.15	-0.052	-9.2	1.07
	Copper - Pure	-0.2	0.209	-0.15	0.052	-9.5	1.119
	Iron - Pure	-0.05	0.051	0	0	-0.05	0.004
Stir-red	Zinc - Pure	-0.05	0.051	-0.05	0.018	-2.7	0.397
	Copper - Pure	-0.05	0.051	-0.05	0.018	-2.7	0.397
	Iron - Pure	0.05	-0.051	0	0	-2.6	0.381

A) Comparing with heavy metals.

(1) Solidification of Zinc

(A) Solidification of Zinc with using sonicator.

The d-value of maximum peak decreased while d-values of the second and the third strongest peak increased. This phenomenon implies that when we solidify zinc with using ultrasonicator, the lattice distance of [100] face decreased and that of [110] and [114] faces increased as zinc ion substituted on calcium ion's sites.

(B) Solidification of Zinc with using magnetic stirrer

The d-value of maximum, the second and the third strongest peak increased. This phenomenon implies that when we solidify zinc with using magnetic stirrer, the lattice distances of [100], [110] and [114] faces increased as zinc ion substituted on calcium ions' sites.

(2) Solidification of Copper

(A) Solidification of Copper with using sonicator.

The d-values of maximum, second and third strongest peak increased. This phenomenon implies that when we solidify copper with using sonicator, the lattice distances of [100], [110] and [114] faces increased as copper ion substituted for aluminium ion.

(B) Solidification of Copper with using magnetic stirrer.

The d-values of maximum, the second and the third strongest peak increased. This phenomenon implies that when we solidify copper with using magnetic stirrer, the lattice distances of [100], [110] and [114] faces increased as copper ion substituted on aluminium ion's sites.

(3) Solidification of Iron

(A) Solidification of Iron with using sonicator.

The d-values of maximum peak and the third strongest peak increased, but the second strongest peak had no change. This phenomenon implies that when we solidify iron with using ultrasonicator, the lattice distances of [100], [114] faces increased and that of [110] face had no change as iron ion substituted on aluminium ion's sites.

(B) Solidification of Iron with using magnetic stirrer.

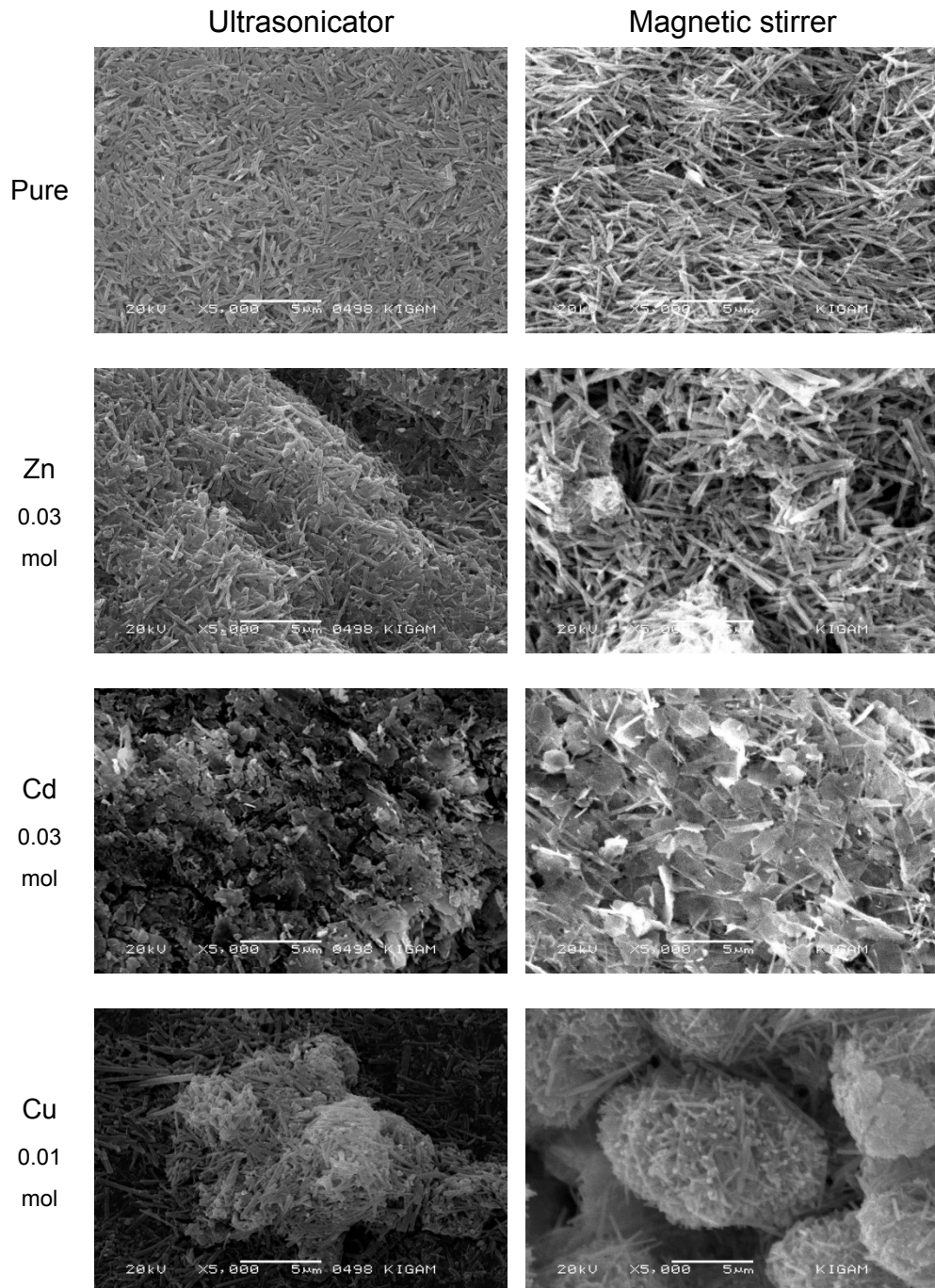
The d-value of maximum peak decreased. The second strongest peak had no change and the third strongest peak increased. This phenomenon implies that when we solidify iron with using magnetic stirrer, the lattice distances of [100] face decreased, that of [110] face had no change, and that of [114] face increased as iron ion substituted on aluminium ion's sites.

(4) Solidification of Cadmium

We got the very new d-value of maximum peak when we solidified cadmium. This implies that new substance were produced, not substituted for calcium or aluminium ion.

The d-value of maximum peak means gypsum, that of second strongest peak means $\text{Cd}_4\text{Al}_2\text{O}_6(\text{SO}_4) \cdot n \text{H}_2\text{O}$.

3) Examination of Ettringite which solidified heavy metals with SEM.



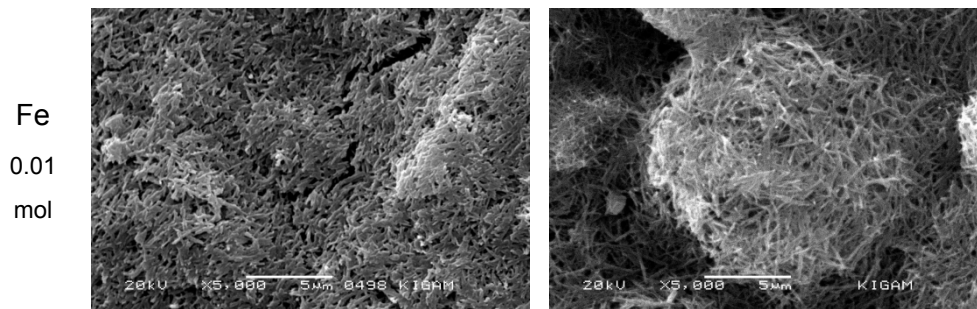


Figure 6. SEM pictures of Ettringites which was synthesized by two different methods and immobilized four heavy metals.

The shape of pure Ettringite is known as a needle-shaped thing. In this study, Ettringites which solidified three heavy metals except cadmium had a needle-shaped crystal, so it was proved that the thing we reacted is definitely Ettringite.

When we tried to solidify cadmium, a new, hexagonal plate-shaped crystal was produced. From this result, we can say that a new substance were produced.

5. Conclusion

A. With using CO_2 , which is one of greenhouse gases, we could eliminate chlorides and stabilize heavy metals. We also synthesized substance of calcium-aluminate system by firing pre-treated bottom ash of 1350°C

B. We could create Ettringite by spontaneous reaction of substance of calcium-aluminate system and gypsum.

C. With ultrasonic method and stirred method, we could synthesize pure Ettringite. Ultrasonic method accelerates the formation of Ettringite. However, too much ultrasonic treatment disturbed the growth of crystal and decompose it physically.

D. Zinc, copper and iron has made substitutional replacements within Ettringite crystals.

E. According to the result of measurement of Ettringite which immobilized heavy metals with substitutional replacements with XRD and SEM, these metals created distortion of Ettringite by being substitutioned and immobilized in Ettringite crystal.

F. Lots of substances shaped hexagonal plate and unreacted gypsums was created in an aqueous solution with Cadmium by way of forming Ettringite. Cadmium is seemed to play a role in disturbing the formation of crystal nucleus and growth of crystal of Ettringite.

G. Injecting CO_2 into the bottom ash, we could get rid of chlorides and stabilize chromium in the material. In other words, we found the key of the prime important matter of recycling bottom ash-heavy metals and chlorides. Also, we could reduce the amount of CO_2 gas simultaneously.

H. With hydration of bottom ash which is treated with CO_2 , we could form Ettringite, new environmental material of new generation.

6. References

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