

BEHAVIOR OF HEAVY METALS IN LANDFILLED FLY ASHES FOR 27 YEARS

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Abstract - Municipal Solid Waste Incineration (MSWI) produces a substantial amount of fly ash (FA) and bottom ash (BA), which contains harmful heavy metals such as Pb, Cd, and Cr. Generally, FA has relatively higher heavy metals content than that of BA. Thus, MSWI FA needs to be pre-treated before landfill disposal. This paper discusses about the behavior of heavy metals in landfilled fly ash by different treatment method in a simulated environment for the past 27 years. X-ray fluorescence (XRF), column experiment, and Japan Leaching Test (JLT-13) were conducted on untreated, chelate treated, phosphate stabilized and slag treated FA. After 27 years, the discussed heavy metals Pb, Cd, Cr, Zn, and Cu from column experiment have stabilized, and the leached Pb and Cd from leaching test fell way below the Japanese regulatory criteria for landfilling of 0.3mg/L. [1] Even though Zn is stabilized, the result of leached Zn is heavily dependent on the pH value of the sample at the time.

1. INTRODUCTION

Due to economic development and population growth, significant amount of municipal solid wastes (MSW) is generated annually in Japan. Incineration has played an important role in solving the scarcity of available land to accommodate the accumulated MSW. Solely in year 2020, 41,670,000 tons of MSW was generated and 79.1% of which were incinerated into 3,640,000 tons landfill waste. [2] MSWI produces a substantial amount of fly ash (FA) and bottom ash (BA), which contains harmful heavy metals such as Pb, Cd, Cr, Zn, and Cu. Generally, FA has relatively higher heavy metals content than that of BA. [3] Thus, MSWI FA fly ash needs to be pre-treated before landfill disposal according to the Waste Disposal and Public Cleansing Law in Japan. [4]

Furthermore, there are far more landfill sites in mountainous areas than in other areas in Japan, and the long-term stabilization of treated fly ash in mountainous landfills has not been clarified. [5] The study investigates the behavior of fly ash from simulated mountainous landfill model tanks for untreated, chelate treated, phosphate treated fly ash, and slag. With chelating treatment being the most commonly used treatment in Japan, XRF, column experiment, and JLT-13 were conducted to evaluate the behavior of fly ash for the past 27 years.

2. MATERIALS AND METHODS

In 2002, two separate research of similar objectives from Fukuoka University were merged into one. [5] [6] Ten landfill model columns were filled with fly ash that has undergone pre-treatment (Table 1). The top end of the column is exposed to natural rainfall, whereas the leachate is collected at the bottom of the column. (Fig.1)

Table 1. Types of ash mix and treatment method corresponding to the column number.

Column No.	Sample Name	Abbreviation
1	Untreated Bottom Ash, Fly Ash, Compost, Crushed Garbage	BAFA CG(U)
2	Chelate treated BAFA CG	BAFA CG(C)
3	Phosphoric acid treated BAFA CG	BAFA CG(P)
4	Untreated Bottom Ash	BA (U)
5	Untreated Fly Ash	FA (U)
6	Untreated Bottom Ash + Fly Ash	BAFA (U)
7	Surface Melting Slag	SMS
8	Plasma Melting Slag	PMS
9	Chelate treated Fly Ash	FA (C)
10	Phosphoric acid treated Fly Ash	FA (P)

The BAFA CG ash mix has a 60:20:15:5 ratio corresponding to BA: FA: Garbage: Compost. BAFA (U) and SMS has a 3:1 ratio for BA:FA whereas PMS has a 4:1 BA to FA ratio. The rest of the samples are purely 100% FA and BA respectively.

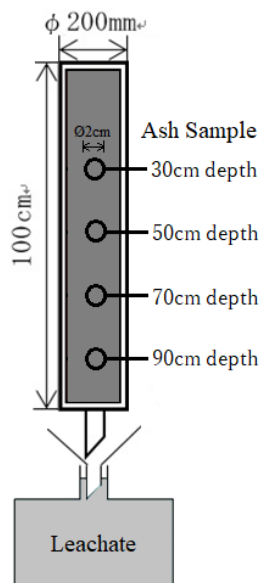


Fig. 1. Column sketch of Lysimeter

Sample Collection

For ash collection, 2cm hole was drilled on 4 collection points, at the depth of 90cm, 70cm, 50cm, and 30cm. (Fig. 1) 80g of ashes were collected from every point. 30g of ash samples were oven dried at 105 °C for 24h to remove moisture for XRF. To prepare for JLT-13, 500mL of super pure water were mixed with 50g ash sample to achieve a liquid to solid (L/S) ratio of 10, and shaken horizontally for 6h at 200 oscillations per minute. The samples were then filtered through a glass-fiber filter, followed by separating 50mL of the filtered sample for analyzing. For column experiment, 500mL of leachate samples were collected from all ten columns. Similarly, the leachate was filtered through a glass-fiber filter and 50mL were separated for analyzing.

Sample Preparation

For XRF test, 10g of oven-dried ash samples were further heated at 600 °C for 2h to calculate the Loss on Ignition (LOI). For column experiment and leaching test, 5mL of 61% nitric acid (HNO_3) was added to the 50mL of filtered sample prepared in a conical flask, which was left to evaporate until 10mL of solution remains using the HPR-4030 hot plate that was pre-heated to 150°C. Another 5mL of 61% HNO_3 was subsequently added to the 10mL solution. Glass lids were placed onto every conical flask before evaporating the solution again until 5mL remains. The 5mL solution was diluted with 1% HNO_3 in a 50mL volumetric flask.

Analytical Method

The chemical composition of MSWI fly ash were determined by using XRF spectrometer (Rigaku RIX3100). 4g of oven-dried ash samples were formed into pressed pellets by pulverization with a 4:1 ash sample to binder ratio.

The concentration of heavy metals leached were determined by using Inductively Coupled Plasma Optical Emission Spectroscopy (720 ICP-OES). The prepared samples were tested against standard solutions of 0.005 mg/L, 0.01mg/L, 0.02 mg/L, 0.05 mg/L, 0.1 mg/L, 0.5 mg/L, and 1 mg/L.

3. RESULTS AND DISCUSSION

Bulk Chemical Composition Analysis

XRF was conducted to determine the constituents present in the ash samples. According to Table 2, the MSWI ash samples comprises mainly of SiO_2 , Al_2O_3 , Fe_2O_3 , and CaO . Interestingly, samples with consisting of only pure FA: FA (U), FA (C), and FA (P) have higher CaO weight percentage (wt%) than that of SiO_2 . Inversely, samples with BA have higher SiO_2 content.

These three trace constituents - Pb, Cu, and Zn particularly displayed significant concentrations. Cd and Cr have a smaller ppm in comparison, but as both Cd and Cr are considered harmful heavy metals, these five trace constituents are selected for this study.

Generally, the amount of insoluble Pb, Cd and Zn present in the 10 samples decreased in year 2000 from year 1996 as shown in Table 3. Contrastingly, apart from BAFA CG ash mix samples, the amount of insoluble Cr increases. Chelate treatment causes the amount of Cu to decrease, whereas phosphate treatment results in Cu to increase. The amount of trace constituent in 2022 has a random pattern when compared to previous results. Some increased drastically, while some showed very little changes. When the two research were merged, only a small fraction of ash were randomly extracted from 8m height by 1m diameter model column and stored in the current column of 1m height by 0.2m diameter. The huge reduction in amount of non-homogeneous ash samples transferred may have resulted in the inconsistent results.

Table 2. Bulk Compositional Analysis (XRF)

Sample Name			BAFA CG (U)	BAFA CG (C)	BAFA CG (P)	BA (U)	FA (U)	BAFA (U)	SMS	PMS	FA (C)	FA (P)
Major Constituent (mass%)	SiO ₂	[%]	25.5	33.9	34.5	29.3	18.7	25.9	33.8	40.2	20.6	11.7
	Al ₂ O ₃		16.2	16.4	16.3	18.6	8.8	17.4	17.7	19.4	10.1	6.5
	Fe ₂ O ₃		7.6	7.7	7.8	17.4	2.1	15.1	11.4	3.6	2.3	1.7
	CaO		23.3	20.9	20.9	19.5	38.0	24.7	26.5	30.3	40.0	33.6
	Sub Total		72.5	78.9	79.6	84.8	67.7	83.1	89.4	93.6	73.0	53.5
Minor Constituent (mass%)	TiO ₂	[%]	1.26	1.26	1.24	1.47	0.79	1.18	1.24	1.34	0.88	0.61
	MnO		0.11	0.10	0.12	0.21	0.07	0.17	0.17	0.17	0.07	0.06
	P ₂ O ₅		2.59	2.86	3.74	1.97	0.72	1.49	2.66	0.16	0.80	25.1
	MgO		2.86	2.79	2.79	2.50	2.17	2.47	2.69	2.56	2.47	1.08
	Na ₂ O		1.30	1.43	1.60	1.09	0.67	0.90	1.85	0.67	0.70	4.54
	K ₂ O		0.52	0.56	0.58	0.37	0.02	0.27	0.48	0.11	0.05	0.23
	Cl		0.13	0.14	0.11	0.08	0.13	0.10	0.24	0.44	0.12	0.70
	S		0.05	0.05	0.05	0.10	1.49	0.14	0.04	0.39	1.13	0.47
	F		0.03	0.03	0.03	0.01	0.19	0.09	0.00	0.03	0.24	0.21
	Sub Total		8.84	9.22	10.26	7.80	6.25	6.81	9.37	5.87	6.45	32.95
Trace Constituent (ppm)	Zn	[ppm]	5865	5034	5497	4511	16188	7444	4650	496	16776	5907
	Cu		2388	2536	1847	3507	822	2342	1341	207	831	650
	Pb		3865	4871	4364	1729	2414	2826	715	49	4130	1941
	Cr		243	228	231	782	304	601	1182	715	294	215
	Ni		103	106	97	311	83	188	162	26	81	77
	Ba		851	791	756	1235	339	1014	1100	1460	468	362
	Sb		0	0	0	106	0	0	0	0	0	1
	Sn		139	157	162	305	289	235	101	49	317	227
	Sr		359	316	324	292	59	251	392	502	93	134
	As		26	33	29	12	16	19	5	0	28	13
	V		3	3	3	4	2	3	4	3	2	2
	Cd		107	102	102	98	174	120	95	79	199	105
	Co		7	6	6	3	1	3	7	13	2	3
	Sub Total [%]		1.40	1.42	1.34	1.29	2.07	1.50	0.98	0.36	2.32	0.96
	LOI (%)		9.8	10.4	8.9	6.1	24.0	8.5	0.3	0.2	18.3	12.6
	Total	[%]	82.75	89.58	91.15	93.93	75.97	91.47	99.75	99.85	81.73	87.45

Table 3. Comparison of Chemical Composition in Ash Samples in Year 1995/1996, 2000, and 2022 (XRF)

No.	Sample	Trace Constituents																			
		Pb (mg/kg)			Cd (mg/kg)			Cr (mg/kg)			Zn (mg/L)			Cu (mg/L)							
1	BAFA CG (U)	1995	2640	2123	3865	1995	25	27.5	107	1995	40	25.0	243	1995	2835	3570	5865	1995	1455	1640	2388
2	BAFA CG (C)		2435	2358	4871		10	25.0	102		40	27.5	228		3030	448	5034		1515	578	2536
3	BAFA CG (P)		2695	2293	4364		25	27.5	102		40	17.5	231		2855	558	5497		1475	1503	1847
4	BA (U)	2000	3100	1750	1729	2000	20	4.7	98	2000	810	1300	782	2000	6000	2950	4511	2000	2300	2500	3507
5	FA (U)		3300	2550	2414		190	110.0	174		270	255	304		17000	10850	16188		650	570	822
6	BAFA (U)		4700	2400	2826		45	26.5	120		680	855	601		6800	4600	7444		4000	3600	2342
7	SMS	1996	610	545	715	1996	9.9	0.2	95	1996	890	1100	1182	1996	4800	4000	4650	1996	2600	1550	1341
8	PMS		210	60	49		0.2	79	430		515	715	410		320	496	130		245	207	
9	FA (C)		2800	2850	4130		180	125.0	199		250	265	294		17000	10800	16776		600	595	831
10	FA (P)		3300	2200	1941		160	125.0	105		210	290	215		14000	7450	5907		470	635	650

Behavior of FA Leachate from Column Experiment

In a simulated environment where the ashes are exposed to natural rainfall, leachate was collected every month for the first 5 years, then annually for the following 22 years. The most common pattern seen in all the graphs below, from Fig. 2 to Fig 5, is that by year 2005, all heavy metals discussed have concentrations of less than 0.5mg/L. Furthermore, leached harmful heavy metals Pb, Cd, and Cr were

already below detection limit by year 2000, hence the consistent overlapping of data points is visible on the graphs from 7 years onwards.

Fig 2a. shows that Pb leached out of BA (U) decreased gradually throughout the 27 years from 0.2mg/L to 0.01mg/L. Initially (Year 1995), 3300mg/L of Pb was leached out of FA (U), which decreased to 1800mg/L in the 2nd year. The concentration of Pb gradually

decreased from then on for the next 4.5 years, before arriving at a constant value of 0.01mg/L up until year 2022 due to the limitations of ICP-OES equipment. As for BAFA (U), the amount of Pb leached was between BA (U) and FA (U) at 1100mg/L in the beginning, before reducing to 0.05mg/L in less than 3 years, which is quicker than FA, as anticipated. As for Cd (Fig. 2b), the samples with raw FA have more leached Cd in comparison to samples without FA. The Cr results from the column experiment shows that only FA (U) had high amount of Cr leached in 1st year at 4mg/L. (Fig. 2c) All the other samples showed very low leached Cr values. (Fig 3c; 4c; 5c)

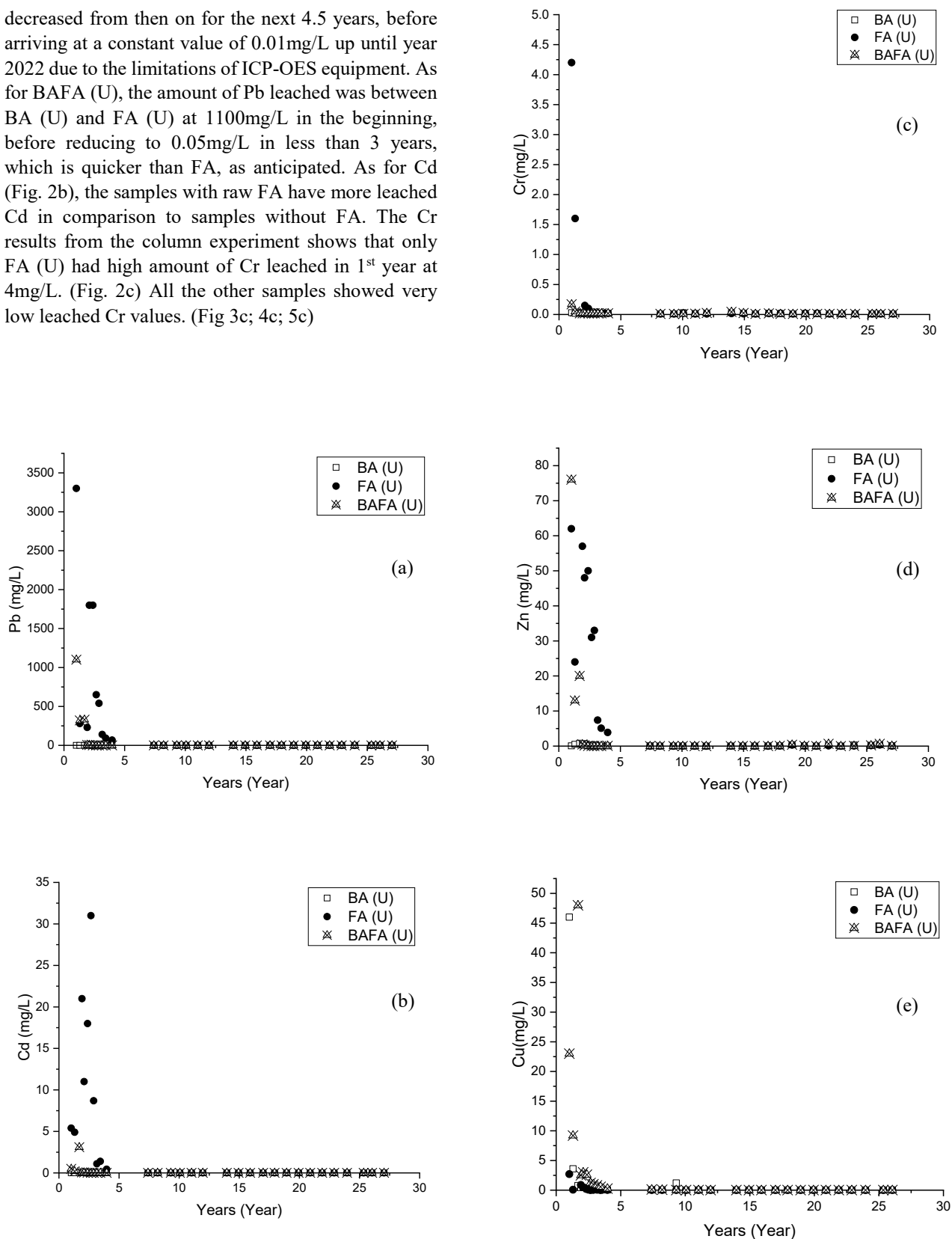


Fig 2. Leached heavy metals for untreated raw BA and FA: (a) Pb; (b) Cd; (c) Cr; (d) Zn; (e) Cu

The leached Pb in BAFA CG mixed ash samples (Fig. 3a) had a peak value of 0.69mg/L, 0.45mg/L, and 0.43mg/L for sample 1 to 3 respectively. As for Cd, there was very little Cd detection throughout 27 years for BAFA CG (U). The leached Pb meets standard landfill criterion of 0.3mg/L in less than a year. [7] Contrastingly, in Fig. 3b, Cd detection was initially low in the first quarter of the 1st year for BAFA CG (C) and BAFACG (P), but the leached Cd spiked to 0.33mg/L and 0.4mg/L respectively before decreasing to less than 0.1mg/L, which remained unchanged until today. This particular ash mix has a larger proportion of BA compared to FA. According to the leached Pb and Cd results (Fig. 2a and 2b), BA has significantly lower leached Pb and Cd value. Thus, the ash mix ratio affects leached concentration of Pb and Cd.

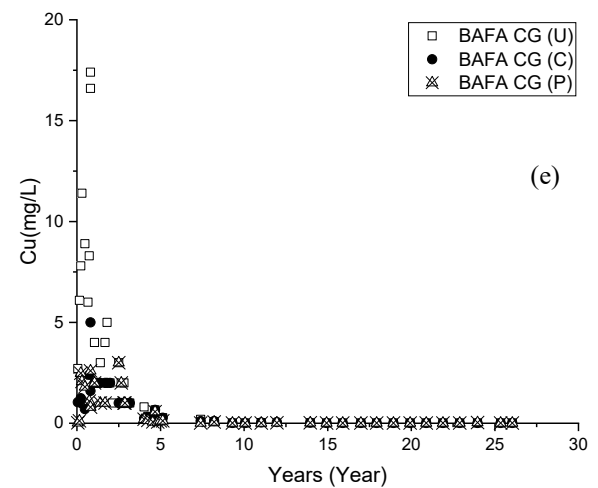
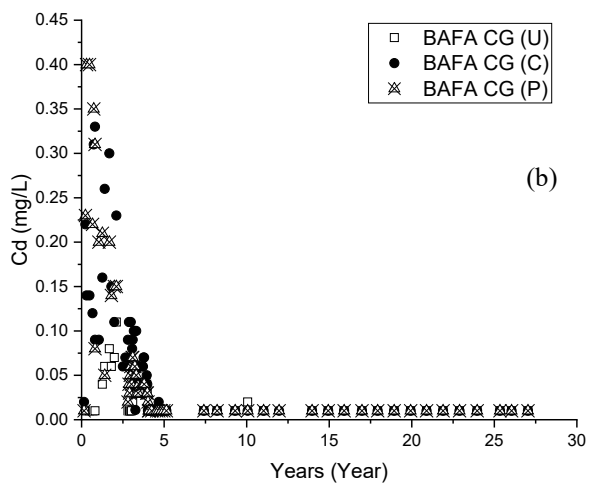
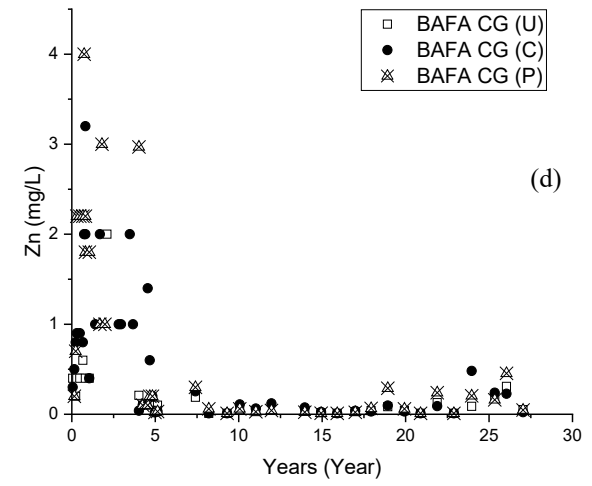
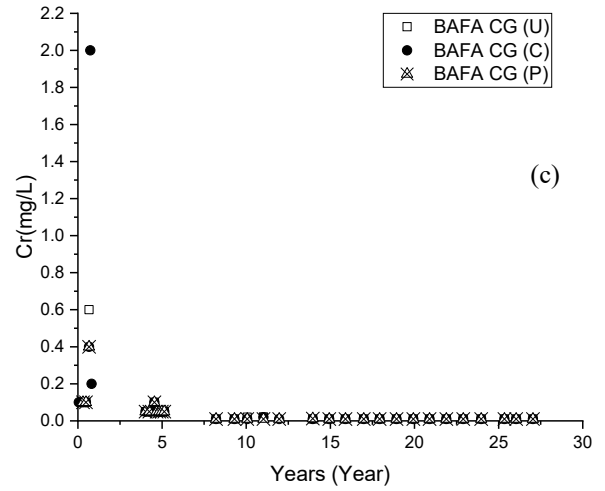
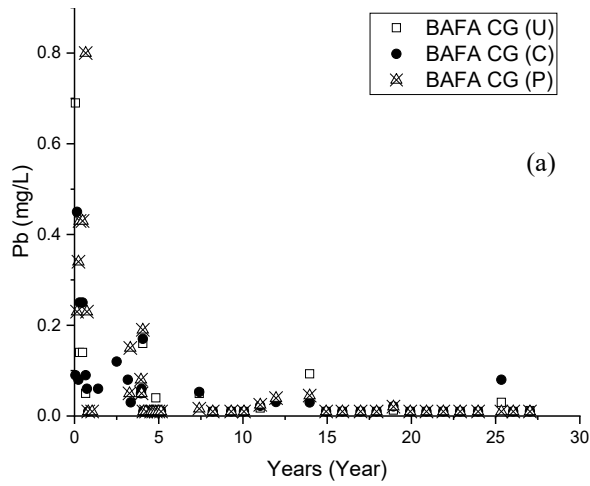


Fig 3. Leached heavy metals for BAFA CG ash mix: (a) Pb; (b) Cd; (c) Cr; (d) Zn; (e) Cu

The samples with slag treatment in this study consists of BA and FA mix. Both SMS and PMS displayed stable and safe amount of leached heavy metals (Pb, Cd, Cr, Zn, and Cu) throughout the 27 years. Most of the leached heavy metals are below detectable limit and does not exceed the concentration of 0.09mg/L as shown in the graphs in Figure 4.

According to the results from column experiment in Figure 4 and leaching test in Table 4, slag treated MSWI FA and BA should be classified as general non-hazardous waste, that meets the effluent standards. In view of the fact that MSWI FA and BA slag will not cause pollution due to the leaching of heavy metals, it is possible to reuse MSWI FA and BA.

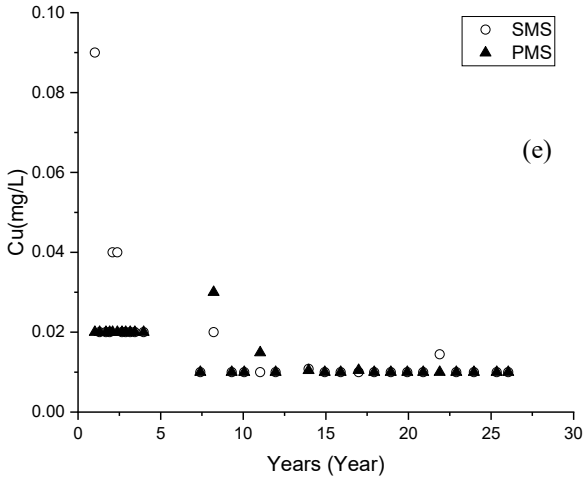
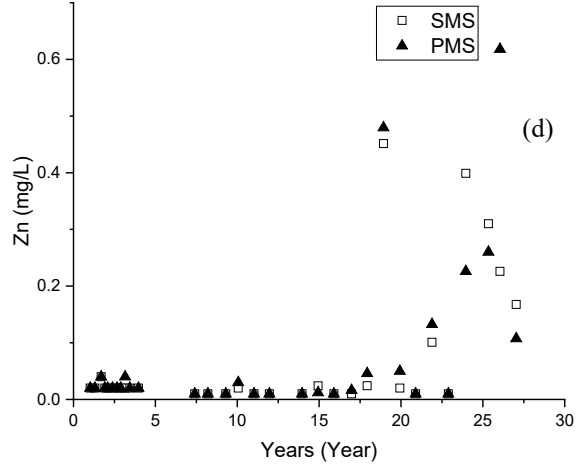
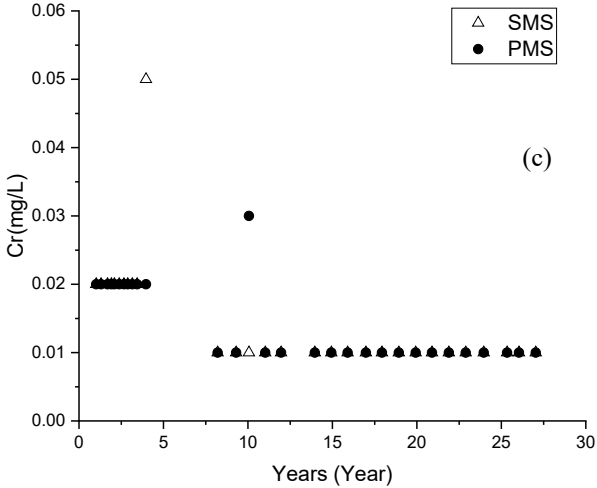
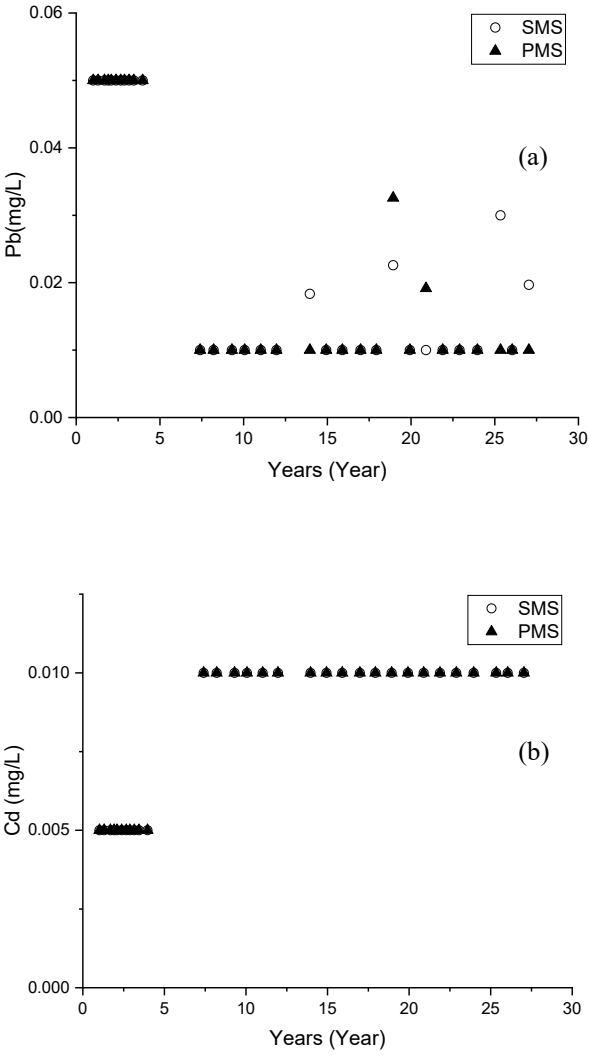


Fig 4. Leached heavy metals for slag treated FA and BA: (a) Pb; (b) Cd; (c) Cr; (d) Zn; (e) Cu

Phosphate stabilization is very effective at reducing the leachability of heavy metals, particularly on Pb, over the entire pH range in comparison to chelate treatment. [1] This is supported by the results in Fig. 5a. FA (P) had a significantly larger leached amount of Pb as compared to chelate treated FA (C) at peak value of 6600mg/L and 110mg/L respectively. However, FA (P) stabilized in approximately 3.5 years, twice the speed of FA (C) which stabilized in 7 years. Similarly for FA (P) on Cd (Fig. 5b), the concentration leached was very high in comparison with FA (C) at 210mg/L, and also stabilized in 3.5 years. Contrastingly, the leached Cd in FA (C) fluctuated between 0.1mg/L 0.3mg/L for 4 years before arriving at a constant value of 0.01mg/L. However, even though phosphate stabilization is a quicker method, chelate treatment displayed a significantly lower leached amount of Pb and Cd and is more consistent and gradual method in comparison.

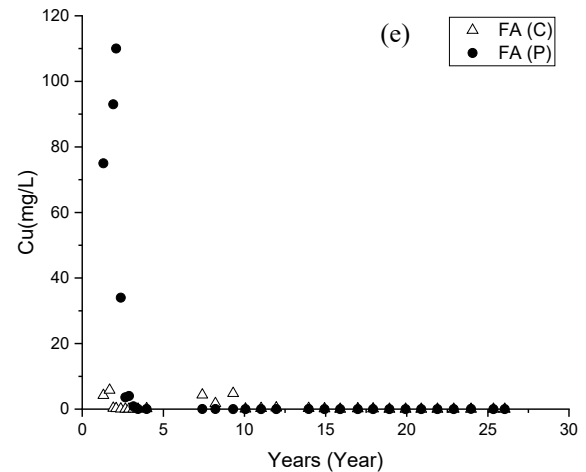
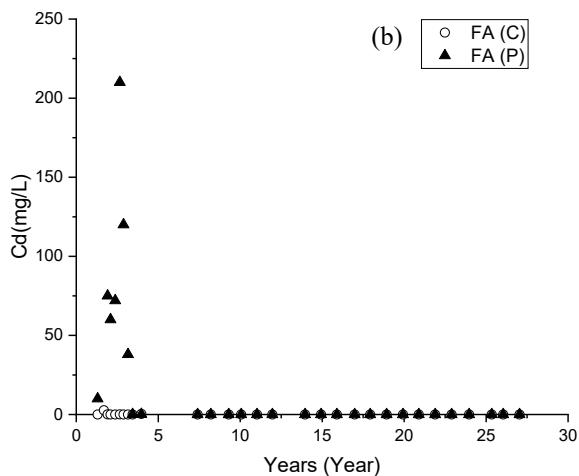
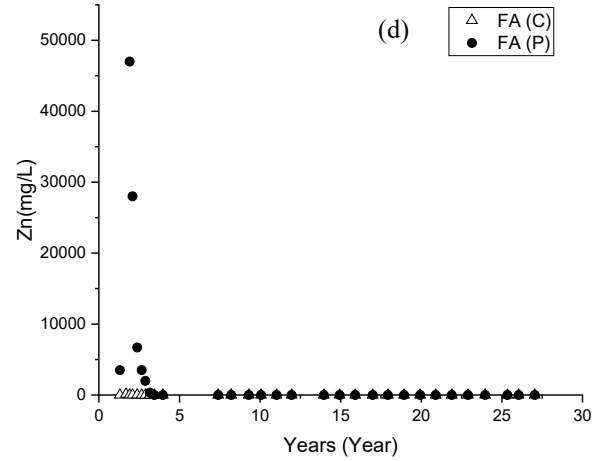
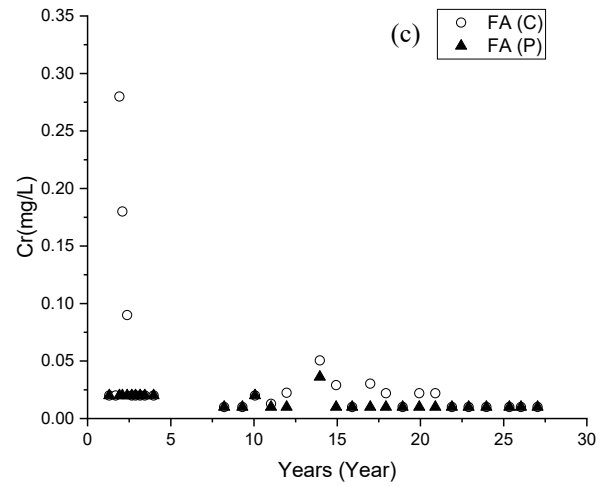
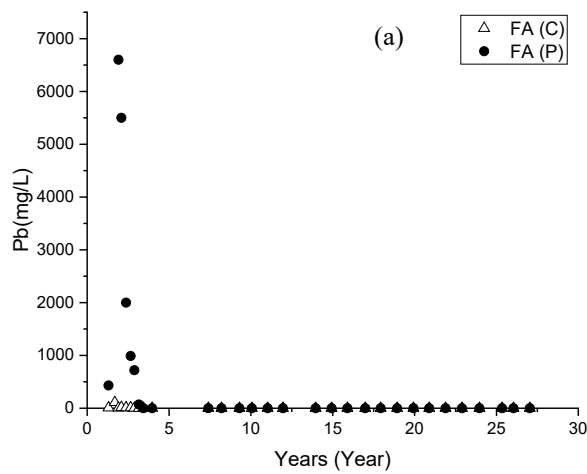


Fig 5. Leached heavy metals for treated FA: (a) Pb; (b) Cd; (c) Cr; (d) Zn; (e) Cu

Leaching Behavior of BA and FA (Leaching Test)

JLT-13 conducted three times: year 1995 for sample 1 to 3, year 1996 for sample 4 to 10, year 2000 and year 2022, as shown on Table 4. The BAFA CG (U), BAFA CG (C), BAFA CG (P), and FA (C) exhibits the trend of increased leached Pb in year 2000 before decreasing to meet the landfill disposal criteria of 0.3mg/L. [7] The increase in amount of leached Pb may be caused by the non-homogeneous ash sample. Contrastingly, BA (U), FA (U), BAFA (U), and FA (P) showed decrement in leached Pb throughout the three tests.

Slag is the only treatment method that displayed stable, safe and consistent amount of leached heavy metals for both leaching tests. Almost all of the leached Cd are

below detection limit. BAFA CG (U), BAFA CG (C), BAFA CG (P) has the same amount of leached Cr in year 1995 and 2000, followed by a below detection limit value in year 2022. Thus, as mentioned in the discussion for the leaching behavior of heavy metals from the leachate, slag treated MSWI FA and BA is safe for reuse.

In year 1996, BA (U), FA (U), BAFA (U), and BAFA CG (P) initially leached relatively high concentration of Cu at 1.50mg/L, 0.26mg/L, 1.50mg/L and 0.40mg/L respectively. These four samples exhibit a decrement trend over time. BAFA CG (U) and BAFA CG (C) on the other hand has an increased in leached Cu in year 2000, while SMS, PMS, FA (C), and FA (P) exhibited

Table 4. Leached Heavy Metals from Leaching Test (JLT-13):

(a) Harmful Heavy Metals (b) Not Harmful Heavy Metals

(a)

No.	Sample	Heavy Metals																	
		Pb (mg/L)						Cd (mg/L)				Cr (mg/L)							
1	BAFA CG (U)	1995	0.06	2000	0.07	2022	0.04	1995	>0.01	2000	0.01	2022	>0.01	1995	0.20	2000	0.20	2022	>0.01
2	BAFA CG (C)		0.05		0.49		0.04		>0.01		>0.01		>0.01						
3	BAFA CG (P)		0.05		0.37		0.02		>0.01		0.02		>0.01						
4	BA (U)	1996	0.36	2000	0.05	2022	>0.01	1996	>0.01	2000	>0.01	2022	>0.01	1996	0.02	2000	>0.02	2022	>0.01
5	FA (U)		95		23.5		0.02		>0.01		>0.01		>0.01						
6	BAFA (U)		6.6		0.05		0.01		>0.01		>0.01		>0.01						
7	SMS		>0.05		>0.05		0.02		>0.01		>0.01		>0.01						
8	PMS		>0.05		>0.05		0.01		>0.01		>0.01		>0.01						
9	FA (C)		0.09		0.16		0.01		>0.01		>0.01		>0.01						
10	FA (P)		0.05		>0.05		0.01		>0.01		>0.01		>0.01						

(b)

No.	Sample	Heavy Metals											
		Zn (mg/L)						Cu (mg/L)					
1	BAFA CG (U)	1995	0.10	2000	0.28	2022	0.05	1995	0.53	2000	0.64	2022	0.03
2	BAFA CG (C)		0.10		0.25		0.05		0.10		0.18		0.02
3	BAFA CG (P)		0.10		0.34		0.05		0.40		0.17		0.02
4	BA (U)	1996	0.05	2000	0.02	2022	0.03	1996	1.50	2000	0.06	2022	0.02
5	FA (U)		9.80		1.11		0.04		0.26		0.02		0.01
6	BAFA (U)		2.20		0.02		0.03		1.50		0.28		0.01
7	SMS		0.02		0.02		0.06		0.02		0.02		0.04
8	PMS		0.17		0.03		0.04		0.17		0.02		0.01
9	FA (C)		0.02		0.58		0.05		0.02		0.02		0.03
10	FA (P)		0.02		6.03		0.03		0.02		0.02		0.01

Table 5. Comparison between In-House Laboratory and External Laboratory Test

Sample		BAFA CG (U)	BAFA CG (C)	BAFA CG (P)	BA (U)	FA (U)	BAFA (U)	SMS	PMS	FA (C)	FA (P)
pH	In-House Laboratory	8.0	8.0	8.2	8.7	11.7	8.9	8.2	8.1	10.1	8.5
	External Laboratory	8.9	8.8	9.0	9.2	11.8	9.0	8.6	8.2	9.6	8.5
Zn	In-House Laboratory	0.06	0.05	0.03	0.03	0.06	0.04	0.05	0.05	0.03	0.03
	External Laboratory	0.025	0.022	0.006	0.005	0.005	0.005	0.039	0.014	0.007	0.005

low leached Cu values. As time elapsed, the leached Cu results of all ten samples gradually shifted towards a low and stabilized range of 0.01mg/L and 0.04mg/L.

To ensure the accuracy of the leaching test results. A set of ash samples were sent to an external laboratory to conduct the same test. After data comparison, all the heavy metals have very similar values except for Zn. A comparison of the pH value and Zn for all ten samples are shown in Table 5.

Fig. 6 is a simulation of the relationship between Zn and pH plotted using data from column experiment on Visual Minteq. In the recent 10 years, the leached Zn from column experiment for all samples have been fluctuating between below detection limit and 1.2mg/L. These irregular fluctuation patterns can be explained by Fig 6. The leached Zn corresponds to the pH value of the sample. These samples have the pH value between 8.0 and 9.0, which is the range where a slight change in pH value will result in a large difference in the amount of Zn leached.

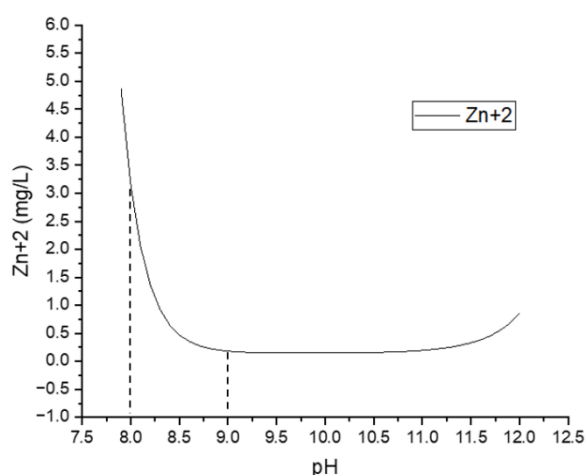


Fig 6. Relationship between Zn and pH

Overall, all heavy metals discussed in the ten samples have either very low leached value or value below the detection limit. Therefore, along with the results from column experiment, it can be concluded that the heavy metals in these ten samples have stabilized.

4. CONCLUSION

In conclusion, leached Pb and Cd from the leaching test fell way below the Japanese regulatory criteria for landfilling of 0.3mg/L. After 27 years, it is safe to say

that the heavy metals in all ten samples are stabilized. Among all the heavy metals tested, the value of leached Zn is heavily dependent on the pH of the ash sample. Among the three treatment methods, slag treatment stabilized the harmful heavy metal the quickest and is the most consistent throughout the 27 years. Phosphoric acid treated FA stabilizes water-leachable Pb and Cd in approximately 3.5 years, which is almost twice as fast in comparison to chelate treated FA that took 7 years. Even though phosphate stabilization is a quicker method, chelate treatment displayed a significantly lower leached amount of Pb and Cd and is a more consistent and gradual method.

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