Bioleaching of fly ash and bottom ash from Municipal Solid Waste Incineration for metal recovery.

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ABSTRACT

PURPOSE

There is a need for an efficient technology that overcomes environmental and financial constraints associated with massive production of solid incineration end-products, i.e., fly ash (FA) and bottom ash (BA) from Municipal solid waste incineration (MSWI). Therefore, a bio-assisted approach has been tested using different experimental setups to obtain energy/cost savings, either by recovering marketable metals or reusing the leach solutions in an integrated system to foster sustainability in the waste industry and other production chains.

METHODS

The leaching of metals from BA and FA was investigated in a controlled lab environment using a culture isolated from a natural system where the dominant strains are acidophilic bacteria, mainly *Acidothiobacillus thiooxidans* and *Acidothiobacillus ferrooxidans*. The effect of different amounts of S^0 and Fe^{3+} was evaluated at two pH set-points.

RESULTS

The bioleaching of BA and FA substrates experienced good yields of metal extraction with an optimum duration of two weeks. The results showed that more than 90% Zn, Cu, and 10% Pb are removed from FA; for the BA samples, the bioleaching resulted in 100% Cu, 80% Zn and 20% Pb removal. Batch experiments with regenerating ion-exchange resins did not perform well for metal recovery, but can suite as a decontamination step.

CONCLUSIONS

The techniques used here with FA and BA can be used for urban mining purposes, but also for low-grade ores in the mining industry, contributing to resource recovery.

Keywords: acidophilic bacteria, MSWI bottom ash, MSWI fly ash, ion exchange resins, resource recovery, Municipal Solid Waste Incineration (MSWI).

1. Introduction

Facing an increasing global demand for metals and the need to minimise the mining and waste management environmental impacts, there is a need to advance metal recovery from wastes, in the context of a circular economy. Million tons of municipal waste are generated every day, leaving massive amounts of metals in landfilled waste and incineration slags. Despite the low absolute concentrations, substance flow analysis on selected Italian municipal solid waste incineration (MSWI) plants shows that fly ash (FA) and bottom ash (BA) can be low concentration streams of high-tech metals, with estimated annual flows of tens of kg and a total content comparable to low-grade active mines [1]. Bioleaching is a reliable hydrometallurgical technology, based on the production of a mineral or organic acid by microorganisms to solubilise metals, and it is widely employed commercially for processing pyrite-rich, low-grade sulphide ores [2-4]. Bioleaching can be a suitable and more sustainable alternative to chemical leaching for resource recovery from wastes.

Bioleaching of Al, Cu, and Zn from MSWI ashes can be suitable for economic recovery using both fungi, mixed acidophilic bacteria [5], and mixed alkaline bacteria [6], but no upscaling of the technology has been developed. Funari et al. [7] demonstrated the leaching behaviour of a wide range of metals that can be removed from MSWI FA using a mixed acidophilic culture in a one-step bioleaching process. The bioleaching procedure (acidic culture medium, 10% pulp density, 150 rpm, 30° C) yielded ~90% Al, Mg, Mn, Zn; ~80% Cu, P, Nd; ~60% Cr, Ga, Ni, Pb; ~50% Ce, Co; 30% La in the leachate solution [7]. Compared to a traditional leaching route (H_2SO_4, 10% pulp density, 150 rpm, 30° C), bioleaching resulted in satisfactory removals suggesting the bio-produced lixiviants as a greener alternative to other solvents [7]. Bioleaching showed low mobility of unvalued elements such as Ca, Si, Fe, and Ti, selective removals of Co, Pb, and REE, with low capital costs. However, there is vast (and relatively unexplored) potential for bioleaching optimisation with limited additional costs, e.g., by improving medium (S^0, Fe^{2+}, nutrients) and substrate quality, thermo-chemical conditions, inoculum volume, and bacterial strains. There is also the need to adapt microorganisms from natural systems, like acidophilic mixed cultures, to tolerate alkaline wastes such as MSWI ashes.

The goal of this study is to investigate the feasibility of biotic leaching of metals from municipal waste incineration fly and bottom ashes by using a mixed acidophilic culture from samples of the Libiola Fe-Cu mine area (Ligurian Apennines, Italy). The effect of different amounts of S^0 and Fe^{2+} was evaluated at two pH set-points to optimise metal recovery. Batch experiments with regenerating ion-exchange resins, Amberlite® IRA-400 vs Chelex®100, were performed to assess the potential metal recovery from the leachate.

2. Materials and Methods

2.1 MSWI samples

The studied materials were collected from two Italian MSWI plants in 2013 following the sampling strategy adopted in Funari et al. [1] for FA and BA samples. The selected incinerators use a thermo-recycling technology with grate-furnaces operating at 950-1050 °C. According to the company yearly report, the total processed waste includes 90% household and 10% special waste, and each incinerator produces around $4 \cdot 10^4$ ton/BA/year and $3 \cdot 10^3$ ton/FA/year as solid end-products. The material pre-treatment included drying at low temperature (40 °C) for one week, grounding, and milling (<40 µm) with an agate vibratory mill disk. The milled BA and FA samples were analysed for the bulk chemical composition (Table 1) and used as the starting material throughout the experiments, except for "RN" BA in the reactor #2 (R2). The sample labelled "RN-R2" was used as raw bottom ash for the batch test to investigate a grain-size that would not require as much mechanical crushing before leaching.

Table 1 – Chemical composition by ICP-OES of the starting materials.

3.	4.	**FE**	**FE7**	**RN**	**RN3**
		MSWI-1 BA	*MSWI-1 FA*	*MSWI-2 BA*	*MSWI-2 FA*
Si	*g/100g*	12.2	7.6	15.1	6.6
Ti	"	0.6	0.5	0.7	0.9
Al	"	3.7	1.8	3.8	3.1
Fe	"	10.6	2.3	6.1	1.0
Mn	"	0.2	0.1	0.1	0.1
Mg	"	2.1	1.2	1.4	1.0
Ca	"	13.8	25.9	20.1	22.0
Na	"	2.4	4.1	2.5	2.0
K	"	0.5	3.4	0.7	1.9
P	"	0.3	0.3	0.5	0.7
S	"	0.5	3.0	0.6	2.9
Cd	*mg/kg*	4.6	177.5	<1	13.4
Co	"	167	27.1	119	28.9
Cr	"	602	313	492	659
Cu	"	13520	2679	3041	355
Ni	"	225	91.6	102	66.6
Pb	"	1136	8688	1475	515
Sb	"	283	1459	141	567
Sn	"	696	1951	498	554
Zn	"	5807	32744	3537	3869

4.3 Microorganisms and culture medium

A mixed culture of iron- and sulphur-oxidising microorganisms was obtained from a natural system by combining several samples from overflows and ponds of the Libiola Fe-Cu mine area (Ligurian Apennines, Italy), collected at the sediment-water interface. The characterisation of the original culture was performed at the FaBiT Department (University of Bologna) using the Terminal Restriction Fragment Length

Polymorphism (T-RFLP) analysis that allows rapid profiling of complex microbial communities based on variation in the 16S rRNA gene. According to the T-RFLP results, the mixed acidophilic culture contained members of *Acidovorax genus*, *At. thiooxidans*, *At. ferrooxidans*, *Chlorobium limicola*, and some species closely related to *Acidothiobacillus* (see SI). For the scope of this paper, accurate profiling of the culture was thought not to be critical because we aim to provide clues for the direct application of naturally available microbial communities in the industrial chain of alkaline waste management and, thereby, engineered bacterial strains tailored for the treatment of MSWI ashes. The pH of the liquid samples representing the original mixed culture varied between 1 and 3, and the samples contained red-brown iron(III) precipitates [8]. The composition of the nutrient growth medium (a modified 9K medium [7,9]) was 3.0 g $(NH_4)_2SO_4$, 0.5g K_2HPO_4, 0.5 g $MgSO_4 \cdot 7H_2O$, 0.1 g KCl, 0.014 g $Ca(NO_3)_2 \cdot 4H_2O$ and 22.5 g $FeSO_4 \cdot 7H_2O$ per litre of deionised water. The modified 9 K medium was adjusted to pH 2.0 with concentrated H_2SO_4. All reagents used were of analytical grade. Details about activation and precultivation of microorganisms are in the SI.

4.4 Scaled-up bioleaching experiments

Each inoculum tailored for BA and FA samples was used in a 1-litre flask containing the corresponding sample. The leaching experiments (one-step bioleaching) were carried out in triplicate using 10% (v/v) MSWI ash and 90% (v/v) bio-lixiviant (containing 90% (v/v) 9K medium and 10% (v/v) adapted inoculum). The flasks were shaken on an incubator shaker (Infors HT Multitron Standard) at 150 rpm at 30 °C for a total duration of 48 days. The bioleaching solution became alkaline after the addition of the solid materials due to the high buffering of the MSWI ashes. Bioreactors were titrated using concentrated H_2SO_4 to different pH starting conditions (3 and 6 pH setpoint) to evaluate a bioleaching process that would not require much chemical pretreatment. The pH and redox potential were measured daily; 10 mL of the slurry was sampled weekly for ICP-OES analysis. Evaporation was corrected gravimetrically by the addition of distilled water. Control tests without inocula were performed in the same conditions. Table 2 reports the experimental set-up where different setpoints (i.e., starting pH) and varied addition of S^0 and Fe^{2+} (as $FeSO_4 \cdot 7H_2O$) were used considering the chemical composition of the MSWI samples (Table 1).

Table 2 – Configuration of the different scaled-up bioleaching experiments.

5.	Type	S^0 [g]	Fe^{2+} [g]	pH setpoint
FE-R1	BA	5.0	11.0	3.0 ± 0.5
FE-R2	BA	5.0	0.0	“
RN3-R1	FA	5.0	11.0	“
RN3-R2	FA	2.5	11.0	“
RN-R1	BA	5.0	11.0	6.0 ± 0.5
RN-R2*	BA	5.0	11.0	“
FE7-R1	FA	5.0	11.0	“
FE7-R2**	FA	5.0	22.0	“

*raw bottom ash (unmilled), **supplemented with 50% (v/v) alkaline inoculum

5.3 Chemical analysis

The analytical determination was done using a Perkin Elmer Optima 5300 DV Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES). A certified reference material (CRM-ES AT-1529, lot number 1319108) was used for quality control. All aqueous samples (10 ml) were filtered (0.45 µm, MCE Membrane Millex HA) with a vacuum pump and preserved with a few drops of HNO_3 (Romil Trace Metal Super Purity Acid, Romil Ltd, Cambridge, UK). For the solid materials (i.e., the starting BA and FA materials), a representative sample of the dried and milled ash was weighed into an Xpress microwave digestion vessel (CEM MARS microwave digestion system, CEM Corporation, Milton Keynes, UK), and 5 mL nitric acid (Romil Trace Metal Super Purity Acid, Romil Ltd, Cambridge, UK) were added. The samples were left overnight to slowly digest at room temperature with the vessel sealed, and vented before microwave extraction. The microwave heating was programmed to heat to 200°C in 15 minutes, then held at that temperature for a further 15 minutes. When cooled to room temperature, the gaseous products from the vessels were vented into a fume cupboard, and then the digests were diluted by weight with pure water (Elga Purelab, 18 Megohm conductivity) into tared 50 mL sample vials. The clear solutions were analysed on the ICP-OES for 69 elements under high argon purge conditions, against a calibration of 10 ppm for all elements. In the batch reactors, pH, Eh, and temperature were also measured daily in a Jenway 3540 pH and conductivity meter.

5.4 Data analysis

Three different kinetic models were fitted to the data. The first one was the shrinking core model theory, where the reaction rate is controlled by solid product layer diffusion [10]. When the leaching rate is controlled by a chemical reaction at the particle’s surface, the process can be represented by a first-order kinetics, a linear plot of gradient k (the first-order rate constant for the surface reaction.). If the leaching process is not affected by a product layer, the amount of reacting material is proportional to the available surface of the unreacted core. When no product layer is formed on the solid phase, the reacting particle would be shrunk during the reaction, finally, the solid phase disappears. For a small particle, this can be explained by a Stokes regime [11].

5.5 Recovery with ion exchange resins

Batch tests were performed with Amberlite®IRA-400 and Chelex® 100 (BIO-RAD) to assess the possibility of using the ion exchange resin for metal recovery. The first is a strong base anion exchange resin with quaternary ammonium functional groups (–N+R3) in a polystyrene matrix, and particle sizes of 600-750 µm. Before use, Amberlite®IRA-400 was converted to the hydroxide form according to Gomes et al. [12,13]. Chelex® 100, classified as weakly acidic cation exchange resin, is a styrene divinylbenzene copolymer with paired iminodiacetate ions that act as chelating groups binding polyvalent metal ions [14]. The hydrated Amberlite resin at a concentration of 50 g L^{-1} was stirred at 150 rpm for 45 min with 100 ml of the leachate. Aqueous samples (10 ml) were taken at 1, 3, 5, 10, 15, 20 and 30 min. After 45 min, the solution was decanted off and sampled for elemental analysis. The resin was then mixed with 20 ml of NaOH 2M and stirred for 30 min to assess metal recovery. The NaOH was decanted off and analysed for elemental analysis. A similar procedure was followed with Chelex® 100, except that the metal recovery was made with 50 ml of 1 N HCl.

6. Results and Discussion

6.1 Characterization of the initial MSWI ashes

The selected BA and FA samples show a variable chemical composition (Table 1). The BA are Ca- and Si-rich materials with significant amounts of Fe and Al; other metals, such as Cu, Pb, and Zn are in the order of g/100g. The FA samples show a relatively less homogenous composition comparing to BA. The FA samples are high in Ca and show variable concentrations of Si and alkaline metals; volatile elements, such as Sb, Sn, and Zn are, at least, one order of magnitude higher than other trace elements. Concerning the presence of metals that may favour the bioleaching, we noted higher amounts of iron (that may be present as Fe^{2+}) and less sulphur (that may be present as nugget of S^0) in BA than FA.

6.2 Scaled-up bioleaching experiments

6.2.1 pH setpoint: 3

For this run of bioleaching experiments, we opted for a relatively high starting pH (setpoint 3.0±0.5 pH) to lessen the environmental impacts and capital costs of the use of mineral acids and, in turn, to deliver a reproducible process. Most of metals are efficiently leached out within 15 days for both BA (Fig. 2) and FA (Fig. 3). The leaching of Zn (and other elements like Sn, Cd, and Ni; see SI) from BA is not significantly affected by the experiment duration. The bioleaching results in 100% Cu, 80% Zn and 20% Pb removal from the BA material. Small changes in metal removals are observed during the BA bioleaching without Fe^{2+}.

The results show that more than 90% Zn, Cu, and 10% Pb are removed from FA (Fig. 3), in an acidic medium supplemented with elemental sulphur and trivalent iron. Halved amounts of elemental sulphur in FA decrease the leaching efficiency of 5-15%. The solubility of Al is particularly enhanced using S^0. The improved performance of a bioreactor fed with elemental sulphur is clearly visible from the curves of pH variation.

6.2.1 pH setpoint: 6

We also tested a higher starting pH (setpoint 6.0±0.5 pH). The bioproduced metabolic substances greatly increase the acidity of the slurry in the BA bioleaching experiment (Fig. 4). This results in efficient removals of Al, Cu and Zn. The latter solubilises mostly in a short period with the optimum efficiency reached in one week. The use of raw BA (RN-R2) produces similar or better yields compared to fine-grained BA (Fig. 4).

This may generate savings because there would be no need of mechanical pretreatment. On the contrary, the bioleaching of FA samples (FE7) did not perform well, neither with the addition of the alkaline inoculum collected downstream in the same mine area (see SI). The pH remained at a steady level during the entire duration of the experiment.

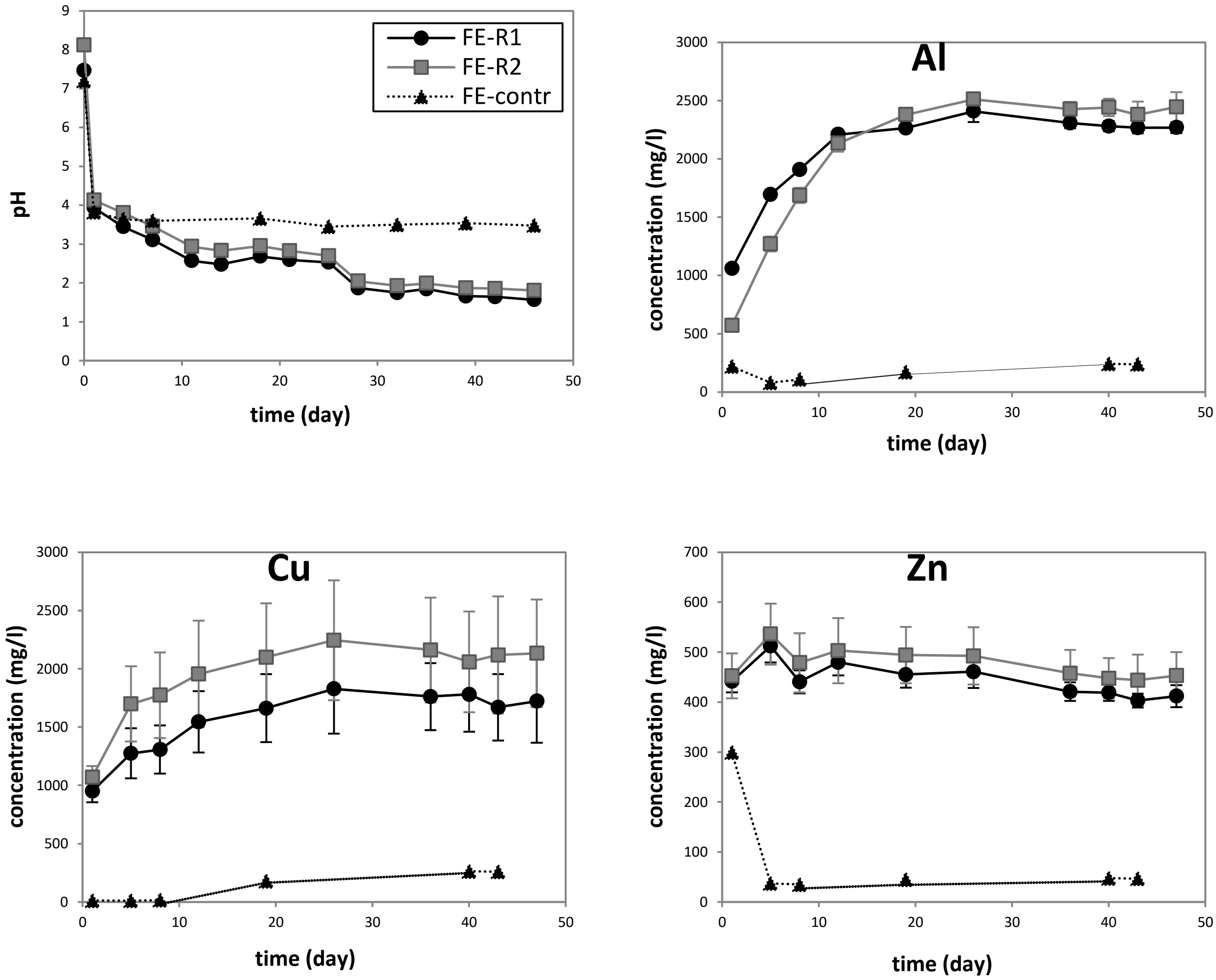

Fig. 1 – Variation of pH (top left) and removals of selected metals as a function of time, during bioleaching of MSWI BA sample from a 3 pH setpoint. Error bars may be smaller than symbols.

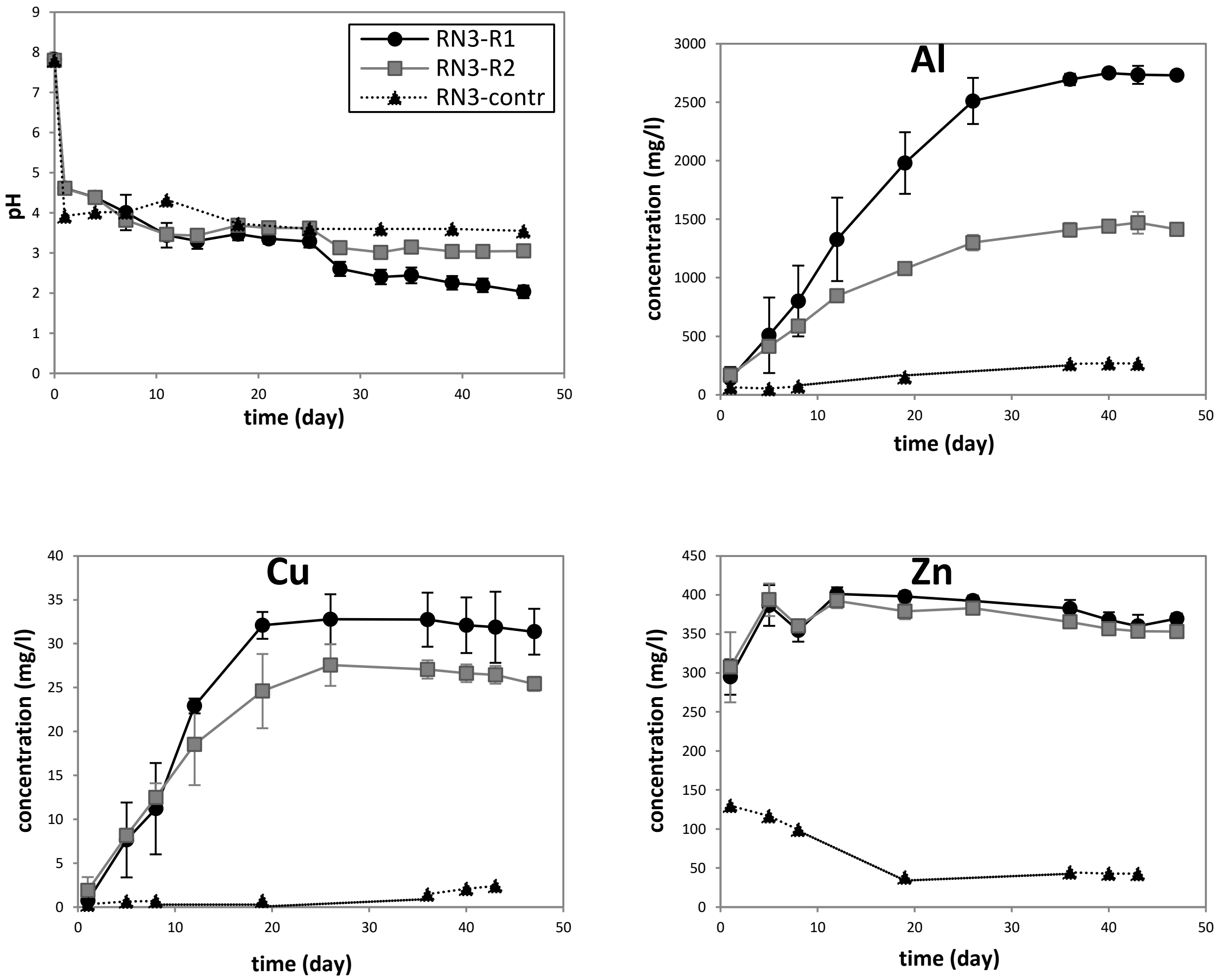

Fig. 2 – Variation of pH (top left) and removals of selected metals as a function of time, during bioleaching of MSWI FA sample from a 3 pH setpoint. Error bars may be smaller than symbols.

3.3 *Kinetics of the bioleaching*

Three different kinetic models were fitted to the results of the experiments (Table 2). FE-R1 and FE-R2 data had better fit the models. Most elements (Al, Cr, Cu, P and Pb) had high coefficient of determination (R2 > 0.95) for the shrinking core model theory (Table 2), which assumes that bioleaching kinetics is controlled by a solid product layer diffusion, and it is usually the best model to explain bioleaching of metals [10, 11, 15]. However, for FE-R1 and FE-R2, cadmium, nickel and zinc fitted better first-order kinetics, which is determined by a chemical reaction at the particle's surface. In general, lower coefficients of determination (R2 < 0.50) were calculated for RN-R1 and RN-R2, and the shrinking core model had better fit to the data. Phosphorus and lead in RN-R1 and RN-R2 fitted the Stokes regime, and Cu the first order kinetics. In all samples, Sn had identical coefficients of determination for both the first order kinetics and the Stokes model, and both models could be used to explain bioleaching.

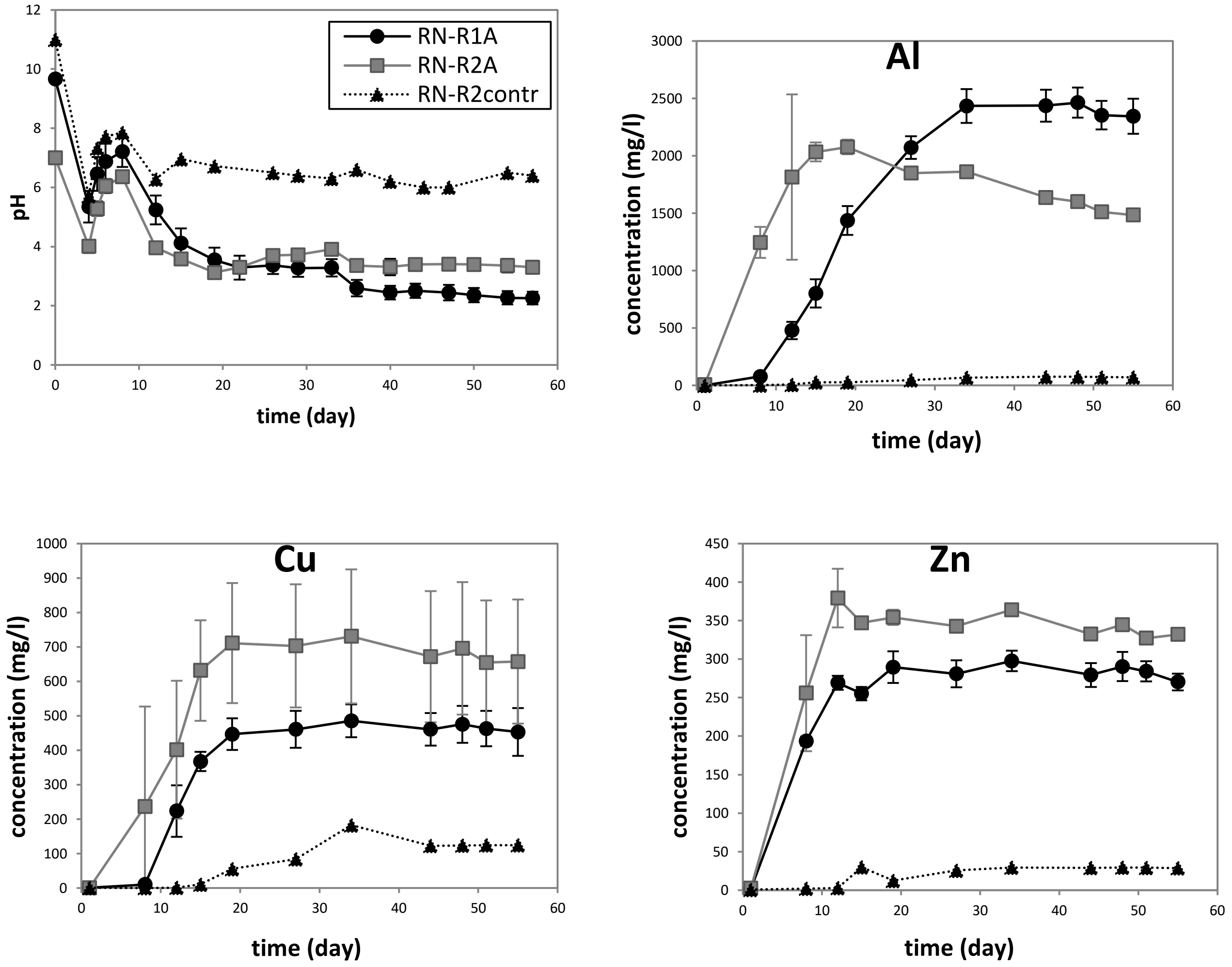

Fig. 3 – Variation of pH (top left) and removals of selected metals as a function of time, during bioleaching of MSWI BA sample from a 6 pH setpoint. Error bars may be smaller than symbols.

Table 3 - Kinetic models applied to the experiments. Equations used for modelling are from Chen et al. [11].

Model	Equation	Substrate	Coefficient of determination (R^2)								
			Al	Cd	Cr	Cu	Ni	P	Pb	Sn	Zn
Shrinking core theory	$k_t = 1 - {}^2/_3 F_t - (1 - F_t)^{2/3}$	FE7-R1	0.782	0.888	0.898	0.983	0.804	0.986	0.973	0.938	0.872
		FE7-R2	0.971	0.884	0.845	0.970	0.690	0.947	0.974	0.926	0.768
		RN-R1	0.864	0.460	0.679	0.606	0.870	0.715	0.955	0.931	0.451
		RN-R2	0.007	0.379	0.982	0.130	0.032	0.863	0.960	0.927	0.322
First order kinetics	$k_t = 1 - (1 - F_t)^{1/3}$	FE7-R1	0.944	0.988	0.850	0.972	0.932	0.963	0.940	0.994	0.976
		FE7-R2	0.951	0.986	0.930	0.964	0.885	0.983	0.910	0.994	0.935
		RN-R1	0.864	0.457	0.871	0.797	0.782	0.901	0.979	0.984	0.419
		RN-R2	0.068	0.371	0.930	0.465	0.491	0.975	0.942	0.982	0.008
Stokes regime	$k_t = 1 - (1 - F_t)^{2/3}$	FE7-R1	0.944	0.977	0.851	0.973	0.929	0.964	0.940	0.994	0.976
		FE7-R2	0.951	0.981	0.930	0.964	0.884	0.983	0.910	0.993	0.933
		RN-R1	0.857	0.451	0.845	0.635	0.734	0.912	0.985	0.984	0.399
		RN-R2	0.082	0.369	0.927	0.130	0.061	0.983	0.963	0.982	0.316

F_t represents the fraction of metal mobilised; *t* is the leaching time, and *k* the rate constant

6.3 Metal recovery with ion exchange resins

The metal-rich leaching solutions were subjected to batch experiments with regenerating ion-exchange resins, Amberlite® IRA-400 vs Chelex®100, to assess the potential metal recovery. Fig. 2 shows the metal recovery for the selected resins. The performances of the two resins were not so encouraging likely due to the presence of competing ions. Nonetheless, around 10% Cu and Pb are recovered by Amberlite® IRA-400. This can be nearly economic considering the substance flow of copper from BA [1]. Alternatively, the use of Amberlite® IRA-400 or another low-priced resin can serve as a decontamination step of the leaching liquor before being recirculated in a closed bioleaching system pushed to the maximum efficiency. The removal of the possible interference anions through selective precipitation could improve the performance of the resin. So further research is needed to minimise interferences and maximise metal recovery.

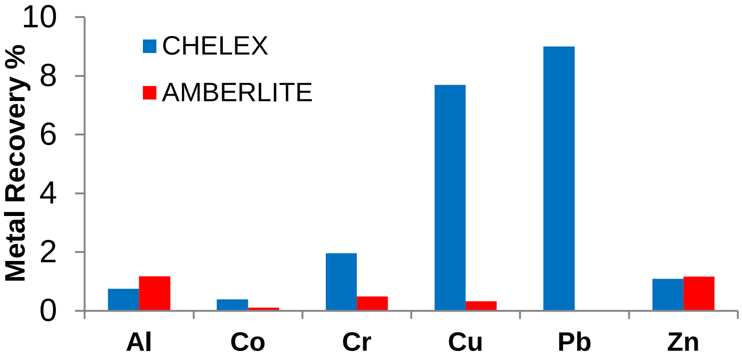

Fig. 4 - Comparison of the ion exchange resins for metal recovery.

7. Conclusion

The results show the potential of bioleaching for mining metals for fly and bottom ashes from municipal solid waste incinerators. The recovery percentage of metals from the leachate using ion exchange resins was not very encouraging, so further developments are needed to separate the metals for recovery and recycling.

Leaching data on MSWI ashes tend to be collected before landfilling (e.g. leaching test) rather than for resource recovery (e.g. total extractable metals), thus a lot of data already collected is of little use for the latter. Nonetheless, MSWI ashes can be a valuable secondary resource if processed in an eco-friendly manner. As such, we reported the results of a bioleaching process with modified experimental setups (Fe^{2+}, S^0, inoculum). The process has the potential to be easily scaled-up and transferred to the waste management industrial chains.

New investments in fine-tuned bioleaching procedures have to be made for secured supply of metals from waste streams. Building-up a knowledge network for the recovery of metals and secondary raw materials from MSWI ashes would be beneficial to make much better use of these anthropogenic resources and thereby to achieve a more circular economy.

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