

## STABILIZATION OF LEAD IN INCINERATION FLY ASH BY AGEING AND CARBONATATION IN CONTACT WITH MOISTURE AND AIR

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### ABSTRACT

Residues from incineration of waste vary considerably in quality not only depending on the composition of the waste and the incineration system, but also on the extent and duration of contact with moisture and carbon dioxide in the atmosphere. Lead has a rather varying abundance and an even more varying availability in ash as determined by leach tests. Fresh fly ash from Jönköping Energi AB has a relatively low content of lead in comparison with other similar ashes but a somewhat high leach rate in relation to the total amount. Thus, in determining the pertinent destinations for this ash, it is appropriate to assess the availability after prolonged contact with moisture and air. It was found that the leaching decreased by up to around three orders of magnitude after such conditioning, which will what take place in a landfill over time. The effect was confirmed by pilot tests. The paper also describes the ash chemistry and possible mechanisms for the stabilization. It is concluded that the stabilization can facilitate landfilling.

*Keywords:* acceptance criteria, ageing, aragonite, ash, calcium carbonate, carbonatation, hazardous waste, incineration, landfilling, leaching, lead.

### 1 BACKGROUND

#### 1.1 Legislation

This paper relates to the Swedish legislation on classification of waste as hazardous or non-hazardous as well as the legislation on acceptance for landfilling.

Classification of waste as hazardous or non-hazardous is to be carried out in accordance with the ordinance of waste [1] which is an implementation in Sweden of the EU waste framework directive [2]. Annex III of this directive has recently been replaced by a EU regulation [3].

Classification as hazardous or non-hazardous waste is to be based on the chemical form, i.e. the inherent properties and the associated hazards. Classification of residues from incineration is thus quite complicated, to a large extent due to the complex chemical constituents. Consequently, methods have been developed in order to make such classifications possible in a practical manner [4, 5].



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Acceptance of waste to landfilling in Sweden is to be carried out in accordance with the regulation on acceptance criteria [6] issued by the Swedish EPA. It constitutes national legislation under the EU landfill directive [7].

The acceptance criteria [6] are based largely on batch and column leach tests. The test methods are applicable and valid only for such waste that does not react with water, and the regulation states that the leach testing should as far as possible and be feasible refer to equilibrium conditions.

In compliance, a method has been developed for ageing of incineration residues under moist conditions, see [4] and references therein.

There is a criterion in [6] that supplements those of leach tests. Thus, waste that has been classified as non-hazardous may be deposited on a landfill for non-hazardous waste without any leach testing. This presupposes that such waste is not co-deposited with such hazardous waste that can be accepted under the leach criteria. This supplementary criterion is not valid in all EU countries.

In practice, local authorities, to whom results of a classification as non-hazardous waste have been presented, may ask about leach data. Moreover, companies themselves may wonder if depositing waste with high leach rates really can be regarded as being compliant with the general statements in our Environmental Code on protection of health and the environment. A key issue in this regard is, of course, to consider if a high leach rate is a short-term effect or if such leaching will take place even after prolonged contact with water and air.

## 1.2 The research question

The situation just described is presently being experienced by Jönköping Energi AB, whose fly ash might be landfilled locally as non-hazardous waste if the leaching of lead could be tolerated. If not, it might have to be treated and eventually deposited in a landfill for hazardous waste.

This situation prompted the present project, which has been financed by Jönköping Energi AB, Miljöhantering i Jönköping AB and Avfall Sverige – Swedish Waste Management and Recycling Association.

Most of the contaminant elements in incineration residues do not constitute such problems as mentioned above for lead. E.g. elements that can have similar ionic radii as iron, e.g. Cr, Ni, V and Zn, are typically incorporated into iron rich phases by solid solution, thus becoming inaccessible to the pore water. [4, 5] Equivalent effects appear also e.g. in some silicates. [4, 5]

Arsenic is, in general, one of the contaminants of high interest from a health and environment point of view. It has been shown, however, that arsenic in ash originates almost exclusively from certain impregnated wood, and that it is therefore feasible to separate arsenic containing waste by sorting using x-ray fluorescence (XRF) equipment [8].

This leaves lead and chlorine as the most potent villains in conjunction with landfilling. The full research question in this paper therefore includes these elements.

## 2 OBJECTIVES AND SCOPE

The objectives and scope of the present paper are as follows.

1. To carry out an information search on how lead might become stabilized in ash during ageing and after contact with water and air.

2. To carry out a similar search with regard to the possibility of removing the chlorides from the ash.
3. To investigate experimentally how the availability of lead may change with ageing in contact with moisture and air.
4. For incineration residues that have been classified as non-hazardous: How disposal at a landfill for non-hazardous waste might be shown to be pertinent also in view of the leaching of lead.
5. For incineration residues that have been classified as hazardous: How disposal at a landfill for hazardous waste or non-hazardous waste might be shown to be pertinent also in view of the leaching of lead and chloride. Possibly, removal of chloride by washing may be much more feasible after (a) lead has been stabilized, and (b) less soluble phases containing chloride have been decomposed. In such a case, the leachant may contain only low levels of the contaminant elements, thus facilitating treatment before release. Similarly, the ash residue may become essentially free from any chlorides.

In addition, some comments will be made regarding the significance (or rather lack of significance) of the speciation of lead for the classification as hazardous or non-hazardous waste.

### 3 INFORMATION SEARCH

#### 3.1 Fuel, incineration and formation of residues

The fuel for the incineration originates from private homes as well as from industry and other professional entities. Pure biofuels can also be included as a part of a fuel mix. The fuel consists mainly of organic matter that is incinerated and thereby converted to gaseous compounds. Most of these are released through the stack, but hydrogen chloride and sulphur dioxide are absorbed using lime (calcium hydroxide) in most cases.

Fuels also contain other constituents that appear as solid residues after incineration. Some of these have passed through the process more or less unchanged, whilst others may have been completely converted to other chemical forms.

The residues thus comprise relatively unchanged inorganic constituents from the fuel, actual ash and spent lime (APC Air Pollution Control residues). Major elements in the residues include O, Si, Al, Ca, Fe, Na, K, Mg and P. Some of the minor elements are of interest in view of their potentially hazardous forms of occurrence, including As, Sb, Pb, Cr, Cu and Zn (contaminant elements). Further information can be found in [9].

#### 3.2 Forms of occurrence, major elements

The residues from incineration appear mostly in oxide form, but some of the cationic elements, especially Ca, Na and K, also appear as chlorides and sulphates.

The properties of a residue can vary considerably depending on the origin (unconverted material, 'true' ash, or spent lime) and the exit point as well as on the ageing in contact with moisture and air. Fly ash, in particular, can be very reactive with regard to water and carbon dioxide, especially its glass phase.

The ageing includes hydration and carbonatation. The alterations are in most cases incongruent. The main phases formed as well as the chemistry in general, are governed by the

composition of the major elements. The pH is usually high and this implies that also aluminium and silicon in oxide form participate in the alterations. Some further information can be found in [9].

### 3.3 Forms of occurrence for minor elements in general

The potential for contaminant elements to cause harm to health and the environment depends largely on the availability to the water phase, and thus also to their forms of occurrence. They have been investigated using a number of tools and the results have been reported in a large number of scientific publications. When such information is to be evaluated and compiled, it is essential to be well aware of the advantages and limitations of the different methods used, including the following.

X-ray diffraction can be used to identify the crystalline phases involved and, thereby, also largely the chemical composition. However, phases in low abundance may be difficult to investigate. The technique is insensitive to amorphous phases and is therefore of little or no use for glass.

Thermodynamic calculations can include even low levels of minor or trace elements, and results can be checked against e.g. leach data. However, thermodynamic equilibrium is assumed. Thus any metastability, e.g. from phases stable at a high but not at a low temperature, cannot be treated correctly. The same can be said about solid solution where none of the commercially available computer codes include such capabilities. It might be added that solid solubility becomes increasingly important the lower the abundance of the element in question. In addition, a case can be made about the degrees of freedom and the number of variables refined.

Electron microscopy does reflect the actual presence of the various elements as well as their distribution among the different phases. However, the sensitivity might not be sufficient to detect a low level of a trace element in phases formed by the major elements.

EXAFS (extended x-ray absorption fine structure) does 'see' all the atoms of an element, and can be used to identify the different types of bonding present. However, the technique is elaborate and expensive, and few investigations have been carried out on incineration residues. Therefore, one frequently has to resort to investigations made on similar materials and elements.

Comparison can also be made with 'model systems' in which the contaminant elements are present at a higher levels, e.g. in natural minerals. A draw back here might be that solid solution may be efficient at low, but not at high levels, cf. the subsequent discussion on lead in section 3.4.

The conclusion after analyzing a large number of publications is as follows.

Any minor element usually appears in more than one chemical form in any given incineration residue. This may or may not include one or more crystalline phases in which such an element is a major one, but it always – or at least in most cases – includes solid solution.

Solid solution usually takes place in the form of substitution that is governed by Goldsmith's rules [10]. They say that substitution can take place when the difference in size of an atom is less than 15% and the difference in charge is less than 2. In addition, the difference in electronegativity should not be too large [10]. The solid solution is driven by the entropy increase on substitution, and the effect is therefore larger, the higher the dilution.

Thus, e.g. Ni, Cr-III, Cu and Zn readily go into solid solution in iron-rich phases [11], [section 6.8.5], Fe being a major element in most incineration residues. Accordingly, and since iron-III (hydr)oxides have very low solubility, these contaminant elements usually have

a low availability to the water phase in aged incineration residues. This includes Cr where the oxidation number III is stabilized by the solid solution [12].

### 3.4 Forms of occurrence for lead

The situation is, however, less straightforward for lead. Only a few of the elements in incineration residues have ionic radii at around  $\pm 15\%$  or less of that for lead, notably Ca, Ba and K. Here, Pb does not associate itself much with K due to the difference in electronegativity, and Ba is not very abundant.

Experimental investigations on fly ash and bottom ash, fresh and aged, have shown, see e.g. [13–23], that lead occurs both as a major element in some phases (minerals) and in the form of solid solution in phases in which calcium is abundant. Lead thus associates itself with calcium as well as iron. The association with calcium includes carbonates, phosphates and ettringite where ettringite may decompose on carbonation to form sulphates and carbonates.

It has been found that carbonation may lead to a decrease of the leaching of Pb by a couple of orders of magnitude, see e.g. [21, 23]. Thus, exposing incineration residues to carbon dioxide may lead to stabilization of any residual lead that might otherwise have been susceptible to leaching, e.g. lead that has been condensed on the ash particles in the low temperature part of a furnace system in the form of chloride [24, 25], or perhaps CaClOH.

It might thus be hypothesized that it is the reaction with carbon dioxide and the stabilization with carbonate that constitute the key to a full stabilization of lead in incineration residues. This is the approach in the present paper for the information search as well as for the experiments. Thus, in the following of the present section, results from surveys of chemical and mineralogical literature are summarized. It is intended that further details, including full references, will be provided in a future journal article.

It is obvious from the above, that for lead, there is a ‘competition’ between the *solid solution* alternative and the *own mineral* one. In this paper *own mineral* refers to situations in which lead is a major element in a phase.

Thermodynamically, it works as follows. At low concentrations in the pore water, lead goes into solid solution with other minerals, but as soon as a solubility product is exceeded lead will go into its own mineral phases, e.g. cerrusite ( $\text{PbCO}_3$ ). Moreover, minor elements may have an impact on which phases are formed with the major elements, thus stabilizing certain phases with a high solid solubility (e.g. aragonite) at the expense of phases with low solid solubility (e.g. calcite). Aragonite and calcite both have the formula  $\text{CaCO}_3$ .

In reality, further factors need consideration. Transport (diffusion) of lead may be limited in relation to the availability of carbon dioxide, in which case own mineral phases are favoured. Formation of new phases such as aragonite may take time, especially with regard to nucleation.

Actually, a number of calcium carbonate phases may form, with or without hydrate water, and the following phases are considered: amorphous calcium carbonate, vaterite, calcite and aragonite.

Amorphous calcium carbonate is formed initially, at least sometimes. It is the most unstable of the four phases considered, and crystallizes to any of the other alternatives. For the phases of ideal  $\text{CaCO}_3$  composition, calcite is the most stable, followed by aragonite and vaterite. The order of stability can be changed by the solid solution, substitution and precipitation conditions, at least for calcite and aragonite.

The ability to pick up lead in solid solution, i.e. substituting calcium with lead, varies considerably with the size of the position for calcium in the structures. Thus, there are nine, eight and six nearest neighbouring oxygen to calcium in the crystal structures of aragonite, vaterite and calcite, respectively. The larger the number of nearest neighbours, the larger the position. Consequently, lead may substitute at room temperature in aragonite at percentage levels while the substitution may be relatively insignificant in calcite.

Substitution can take place in calcite too, but in this case it is ions that are smaller than calcium that substitute well, especially Mg and Fe. Consequently, calcite types of crystal structures with Ca, Mg and Fe as major elements may constitute sinks for minor elements similarly to other iron rich phases.

There is a further important difference between the solid solution and own mineral alternatives. In the latter case, the lead content is about the same throughout the grains formed on dissolution and precipitation. However, with solid solution, lead might be drawn out of solution such that the content becomes highest in the middle of the grains, and very low at the surfaces. This may give rise to leach rates that are much lower than those for the cases of own minerals.

### 3.5 Forms of occurrence for chloride

It is frequently assumed that the element chlorine is easily extracted during leaching in the form of chloride. This might not be entirely true, and it has been reported [26] that some of the chlorine can be bonded into less soluble phases e.g.  $3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{CaCl}_2\cdot 10\text{H}_2\text{O}$ , and  $3\text{CaO}\cdot\text{Fe}_2\text{O}_3\cdot\text{CaCl}_2\cdot 10\text{H}_2\text{O}$ .

Such phases are broken down on carbonatation, in which case the chloride ions are liberated and can be removed with the wash water.

### 3.6 Conclusions for the approach for the experimental work

The information search has shown that carbonatation can lead to a substantial reduction in the leach rate for lead and to a more or less complete leaching of the chlorides. However, there are many different chemical reactions that take place in incineration residues after contact with moisture, carbon dioxide and oxygen, and there may be several reasons for stabilization.

The search has also clearly indicated that such conditions that favour solid solution should be sought. Thus, carbon dioxide should be added at a slow rate and at a low concentration. Diffusion processes should be supported by assuring an open porosity, but yet there should be sufficient pore water to facilitate dissolution and precipitation. Consequently, this constitutes the basis for the selection of the conditions for the experiments.

## 4 EXPERIMENTAL

Three types of tests were performed: can tests, column tests and pilot scale tests. All tests were carried out using fly ash from the Jönköping Energi AB incineration and district heating plant. Its elemental composition is shown in Table 1. The laboratory tests were carried out using granules in the size range 2–4 mm, and the pilot scale tests were conducted using ash mixed with water. The leach properties were evaluated using batch tests, similar to those prescribed in [6], with a liquid to solid ratio of ten, and with a contact time of 24 hours.

The can tests were carried out primarily in order to determine the feasibility and appropriate conditions for the column test. The following parameters were varied: initial moisture

content, time and contact with air. The last was varied by keeping some cans closed, others open and yet others kept open for a couple of minutes every other day. All cans were tumbled for about one minute every other day. A total of 18 such tests were made.

The column test was carried out using 328 g of granules having a water content of 23%. This material was put into a column having a width of 35 mm. Ambient air was sparged into a wash bottle after which it was led through the column. The wash bottle was kept at a temperature somewhat lower than the ambient so that the relative humidity of the air entering the column was about 54%. The flow was around 230 mL/min. The outgoing air was monitored for moisture and carbon dioxide. The arrangement is shown in Fig. 1.

The still ongoing pilot (June 2016) tests are being conducted as shown in Fig. 2. The idea was to test the carbonatation on a sufficiently large scale and under varying wet and dry conditions, as governed by the changing weather conditions.

Table 1: Elemental composition of the fly ash used in the present work. The major elements are figured as oxides.

Major elements	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	Fe <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	MgO	Na <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>
Abundance, wt-%	10.3	3.5	32	2.1	1.81	1.45	3.7	0.54
Minor elements	Sb	As	Ba	Pb	Cu	Cr	Zn	Cd
Abundance, ppm	757	50	1050	1950	630	229	8620	83.1

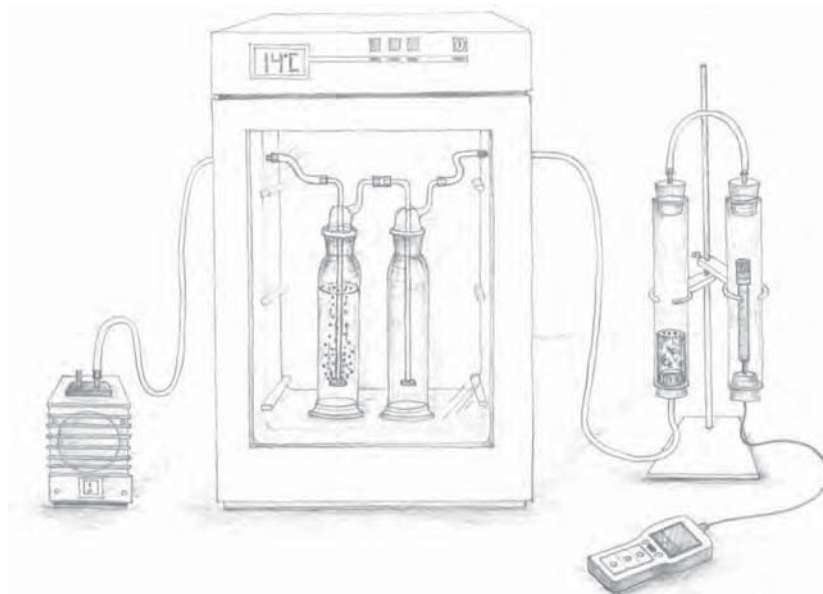


Figure 1: The experimental arrangement for the column test, see text for details. (Illustration drawn by Fabian Sjöblom.)



Figure 2: One of the pilot scale tests, see text for details.

## 5 RESULTS

The results of the can tests were roughly as follows. The initial leach rate for lead for a fresh sample was around 750 mg/kg ash. The moisture content had only a moderate effect. The leach rate for closed cans decreased to around 550 mg/kg after 23 days, and to around 350 mg/kg after 174 days. The corresponding figures for incidentally opened cans were around 525 mg/kg and 300 mg/kg. For constantly open cans, the corresponding figures were around 300 and 1.5 mg/kg, respectively. Thus, carbonatation had a profound effect, while self-curing and oxidation had moderate and inadequate effects. For other ashes studied by one of the authors (Sjöblom), the effect of self-curing with only moisture was found to be much greater, in comparison.

The results from the can tests prompted the initiation of the column test. It was conducted for 39 days with the flow and moisture data given above. The leach rate for lead for the half of the material closest to the air inlet to the column was 0.65 mg/kg, and that for the upper half was 0.89.

Data so far (April 2016) from the pilot scale tests indicate leaching after 14 months days of 46 mg/kg at near the surface and 151 mg/kg at 0,4 m below the surface.

Actually, the results include data for a number of elements, but that will be analysed and presented later.

## 6 CONCLUSIONS, LIMITATIONS AND COMMENTS

The information search showed that lead in incinerator fly ash can form phases in which lead is a major element as well as be included in the form of solid solution in carbonate and other phases formed by the major elements. The solid solution alternative is preferable in view of its more efficient stabilization. Solid solution can be favoured by time, low concentrations and good transport (diffusion).



The experiments have shown that the leaching of lead from fly ash from the incinerator at Jönköping Energi AB will decrease with around three orders of magnitude after contact with moisture and air, and may thus, with a large margin (around an order of magnitude), meet any requirements on leaching for disposal at a landfill for non-hazardous waste. This can be useful in conjunction with ash that has been classified as hazardous as well as non-hazardous waste (see sections 1.1 and 2 above).

It might be tempting to conclude that the stabilization of the present ash on carbonation is due to the formation of solid solutions of lead in carbonates. Although this is clearly what is indicated by the present results, full proof is not provided. Such proof might be obtained, e.g. by electron microscopy in combination with EDX (Energy Dispersive X-ray spectroscopy) and x-ray crystallography. It is not possible to resolve this issue using ordinary thermodynamic calculations, since the commercial programmes available do not include any features that allow calculations on solid solutions.

Another limitation of the present results is that they cannot be used, at least not directly, for the classification of waste as hazardous or non-hazardous. This is because the legislation on classifications of waste as hazardous or non-hazardous lumps together all lead compounds, except for a few that are listed, to certain codes. However, tests may still be carried out in accordance with the legislation [1–3].

Many efforts are presently taken worldwide to wash ash and thereby obtain a material that can either be used at a landfill or deposited on it in a less elaborate manner. The present results indicate that it may be favourable, at least in some cases e.g. when contaminant elements are to be returned to the ash, to first age the ash, and then do the leaching since this will give rise to a better separation of lead and chloride.

Finally, the results indicate that in situ carbonation might be preferable in comparison to one in a chemical process [26]. In situ carbonation has been practiced on an industrial scale for decades in order to improve the Na/Ca ratio in montmorillonite structures and thereby also the swelling properties of bentonite clays.

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