RESOURCES RECOVERY FROM INCINERATION ASHES

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ABSTRACT

Incineration reduces the volume by eliminating the organic content and the potential energy can be utilised. The resources remainning after incineration are the nutrients and the inorganic material. The main component is silicate and aluminium and iron oxide depending on whether aluminium or iron salt is used as precipitation chemicals in the treatment plant. Of the nutrients in ash phosphate is the most important to recover, which can be leached and recovered by acid. Incinerated sludge and ash from co-incineration with solid waste and biofuel were leached with hydrochloric acid at different concentrations and contact time. Leaching with 1 M HCl or higher concentrations and during 8 hours gave more than 75 % dissolution of phosphorus. Metal contents of ash from the co-incinerations and in the leachate were measured and the ratio between metal and phosphorus was calculated. The ratio was higher for ash and leachate than for the sludge. For almost all metals the ratio for ash was higher than the limit for sludge approved for agricultural use. Bottom ash from the grate oven had lower ratios than ash from the fluidized bed reactor. Especially metals with low boiling temperatures were removed from the bottom ash of a grate oven. However, the quotient of metal to phosphorus was higher for ash and leachate than for the sludge and also higher than for sludge approved for agricultural use. Metals, especially cadmium and mercury, can be removed if the metals are evaporated at incineration and phosphorus is recovered from the bottom ash. Chlorine may make chromium voilatile at 500 ºC. To avoid deposition the inorganic material remaining after incineration can be used for production of building materials. The ash can be mixed with cement or concrete, brick or some other object can be made of ash or the ash can be melted and solidified as a ceramic material.

KEYWORDS

Phosphorus recovery, incineration, sewage sludge, building material

INTRODUCTION

Sustainable handling of municipal waste and sewage sludge has as an important goal to recycle resources without supply of harmful substances to humans or the environment (Hultman and Levlin, 1997 and Hultman et al., 1997). Another important goal is to avoid or reduce the amount of waste and sludge that has to be deposited on landfill. In Sweden a tax of 250 SEK/ton on all deposited solid waste is introduced from year 2000 (SFS 1999:673) and deposition of organic material will be prohibited from year 2005 (SFS 1998:902). Anaerobic digestion eliminates half of the organic content and half of the energy can be utilised as methane gas. Incineration is a method that reduces the volume by eliminating the organic content and the potential energy can be utilised (ATV, 1997 and Weibusch et al., 1997). The resources in sludge and solid waste remaining after incineration are nutrients and the inorganic material. Development of method to recover these resources from incineration ashes will make handling of municipal waste and sewage sludge more sustainable.

Mono-incineration of municipal wastewater treatment sludge is not used at present in Sweden. Co-incineration of sludge has been studied by the Stockholm Water Co. (Haglund et al., 1997). Two incineration plants were selected for full-scale co-incineration studies with sludge. In Högdalen incineration plant, municipal solids wastes from Stockholm are incinerated in a grate oven and in Igelsta incineration plant,

biofuels are incinerated in a fluidised bed reactor. Experiences from the co-incineration experiments at the Högdalen plant are reported by Älvesand (1998) and at the Igelsta plant by VAI VA-Projekt AB (1998).

To burn solid waste or sludge different technologies may be used such as grate oven or a fluidized bed. At incineration both bottom ash and fly ash is produced. The fly ash follows with the combustion gases and can be separated with a zyklon. The elements that are evaporated follows with the combustion gases and is separated on the fly ash when the combustion gas is cooled (Bäverman 1997). The fly ash consists of particles on which the evaporated elements in the combustion gas is condensed (see Figure 1). The fraction of the non evaporated material that goes to the fly ash depends on the size of the particles that follows with the combustion gases. More fly ash and less bottom ash is produced when the size of the particles that follows with the combustion gases increases. The share of evaporated and condensed material in the fly ash increases when the amount of fly ash compared to bottom ash decreases. In a fluidized bed reactor the combustion of the sludge occurs in contact with hot sand which is fluidized in a stream of air. Thus, the air stream is much larger than in a conventional oven, and the share of fly ash is much larger.
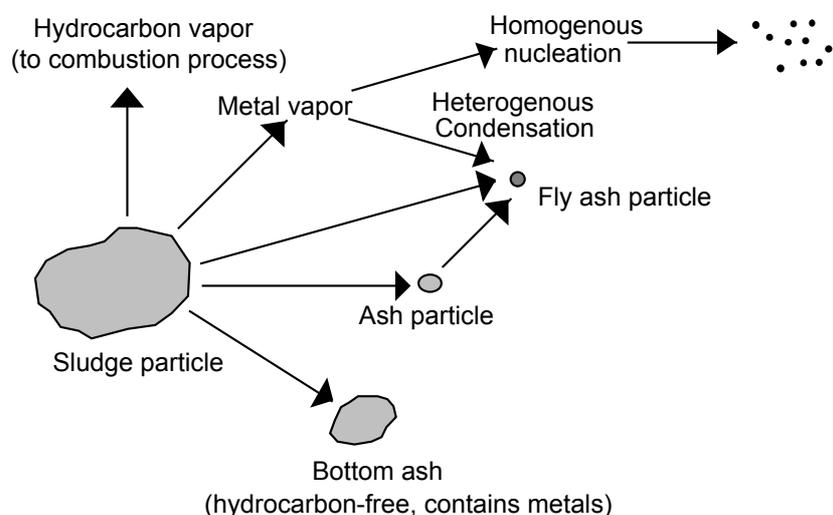
Figure 1. Mechanistic depiction of the fate of trace metals from combustion of sludge (Tillman, 1994).

The inorganic content of sludge from water treatment and wastewater treatment and thus the content in incineration ashes depends mainly on the chemicals used for coagulation of suspended particles in the raw water (Cornwell and Westerhoff, 1981) or for chemical precipitation of phosphorus. Table 1 shows the large difference of the contents in examples of ashes found in litterature. The main component is SiO_2 which originates from soil particles from storm water run off. The Al_2O_3 and Fe_2O_3 may also be high depending on whether aluminium or iron salt is used as precipitation chemicals in the treatment plant.

Reference Element %	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	Na_2O	P_2O_5	SiO_2
Endo et al., 1997	15.6	6.2	7.3	0.6	2.3	0.4	9.2	51.9
Endo et al., 1997	26.0	8.6	5.0	0.7	2.1	1.2	23.9	26.6
Haglund et al., 1997	7.2	5.3	30.0	0.92	1.7	0.54	19.0	19.0
Ozaki et al., 1997	15.6	12.3	5.4	0.5	2.5	0.3	14.0	41.6
Ozaki et al., 1997	14.8	7.9	11.9	1.7	2.8	0.6	20.7	34.6
Ozaki et al., 1997	15.3	10.9	5.5	1.9	3.8	0.4	26.7	31.8
Ozaki et al., 1997	11.1	7.6	24.6	0.8	3.3	0.7	22.1	25.7
Nomura, 1998	22.7	8.2	6.6	2.3	2.4	1.4	1.5	56.3
Takaoka et al., 1997	12.9	17.1	11.2	2.1	2.6	1.1	15.8	30.4
Weibusch and Seyfried, 1997	14.2	10.0	17.9	1.2	1.5	0.7	1.5	36.2
Weibusch and Seyfried, 1997	12.3	13.2	15.1	1.4	1.5	0.6	13.2	36.5
Weibusch and Seyfried, 1997	16.2	20.8	2.8	1.4	1.4	0.6	18.4	30.3
Weibusch and Seyfried, 1997	16.9	16.9	5.6	1.6	1.6	0.7	13.8	35.2

Table 1. Content of different element in examples of ashes from incineration of sewage sludge.

PHOSPHATE RECOVERY FROM ASH

The nutrients in sewage sludge and solid waste are potassium, calcium, phosphate and nitrogen. Of these nutrients phosphate is most important to recover. Phosphate fertiliser is produced by mining of phosphate ores. More than 300 different phosphate minerals are available, but only apatite (calciumphosphate, $Ca_3(PO_4)_2$) is used for production of fertiliser (Corbridge, 1995). In 1995 the world phosphate rock production was 160 000 ton per year (as P_2O_5), having tripled over the last 40 years. About 90% of this is used as fertiliser. At this rate of consumption the known apatite reserves have been estimated to last for a period up to 1000 years. However, if the present increase in world population and the increasing need for fertiliser for food production is taken into account, the supply of phosphate may well be crucial within a century.

Since the concentration of phosphate decreases at lower pH-values, phosphate compounds are soluble in acids and can thus be leached and recovered from incineration ashes by use of acid.

$$H_3PO_4^0 \longleftrightarrow H_2PO_4^- + H^+ \qquad\qquad pKp_1 = 2.148$$
$$H_2PO_4^- \longleftrightarrow HPO_4^{2-} + H^+ \qquad\qquad pKp_2 = 7.198$$
$$HPO_4^{2-} \longleftrightarrow PO_4^{3-} + H^+ \qquad\qquad pKp_3 = 12.023$$

The equilibrium concentration of phosphate is calculated by the formula:

$$[PO_4^{3-}] = K' * [\Sigma PO_4]$$

there: $[\Sigma PO_4]$ = total concentration of phosphate in the solution

$$K' = \frac{1}{1 + 10^{(-pH + 12.023)} + 10^{(-2*pH + 12.023 + 7.198)} + 10^{(-3*pH + 12.023 + 7.198 + 2.148)}}$$

From this relationship the solubility of phosphorus compounds can be calculated. Table 2 shows solubility constant and equilibrium concentrations as a function of K' at dissolution of different phosphate compounds.

Table 2. Solubility constant, Ks (Corbridge, 1995) and equilibrium concentrations as a function of K´ at dissolution of phosphate compounds.

Phosphate compound	Solubility constant, Ks	Equilibrium concentration (mol/litre)	
$Ba_3(PO_4)_2$	$3.4*10^{-23}$	$2.52*10^{-5}(K')^{-0,4}$	
$Mg_3(PO_4)_2$	$6.3*10^{-26}$	$7.14*10^{-6}(K')^{-0,4}$	
$Sr_3(PO_4)_2$	$4.0*10^{-28}$	$2.60*10^{-6}(K')^{-0,4}$	
$Ca_3(PO_4)_2$	$1.4*10^{-29}$	$1.33*10^{-6}(K')^{-0,4}$	$\sqrt[5]{\dfrac{Ks}{(1,5)^3 * (K')^2}}$
$Ni_3(PO_4)_2$	$5.0*10^{-31}$	$6.83*10^{-7}(K')^{-0,4}$	
$Zn_3(PO_4)_2$	$9.0*10^{-33}$	$3.06*10^{-7}(K')^{-0,4}$	
$Co_3(PO_4)_2$	$1.6*10^{-35}$	$8.61*10^{-8}(K')^{-0,4}$	
$Pb_3(PO_4)_2$	$8.0*10^{-43}$	$2.99*10^{-9}(K')^{-0,4}$	
$AlPO_4$	$5.8*10^{-19}$	$7.62*10^{-10}(K')^{-0,5}$	$\sqrt[2]{\dfrac{Ks}{K'}}$
$FePO_4$	$1.3*10^{-22}$	$1.14*10^{-11}(K')^{-0,5}$	

The concentration of for instance calcium phosphate in a solution in equilibrium with calcium and phosphate ions can be calculated from that dissolution of X mole phosphate and gives a dissolution of 1.5 X mole calcium:

$$[PO_4^{3-}]^2 * [Ca^{2+}]^3 = (K' * [\Sigma PO_4])^2 * [1,5\ X]^3 = (K')^2 * (1,5)^3 * [X]^2 * [X]^3 = 1.4*10^{-29}$$

Which gives:

$$X = \sqrt[5]{\frac{1,4*10^{-29}}{(1,5)^3 * (K')^2}} \ \text{mol/litre}$$

However then calculating the solubility or iron and aluminium phosphate consideration has to be taken to iron and aluminium hydroxide precipitation at higher pH-levels.

$$FePO_4\ (s)\ +\ 3\ OH^- \longrightarrow Fe(OH)_3\ (s)\ +\ PO_4^{3-}$$

$$AlPO_4\ (s)\ +\ 3\ OH^- \longrightarrow Al(OH)_3\ (s)\ +\ PO_4^{3-}$$
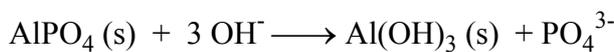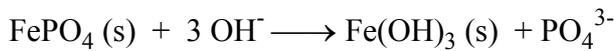
Figure 2 shows the influence on the solubility of iron and aluminium phsophate by hydroxide precipitation. The solubility is lowest around pH-level 6 above which increases with it increasing pH-level. If the phosphate is bound to the ash as iron or aluminium phosphate leaching can be done with base. However, the base used for leaching must be chosen so that dissolved phosphate is not precipitated by the cations.
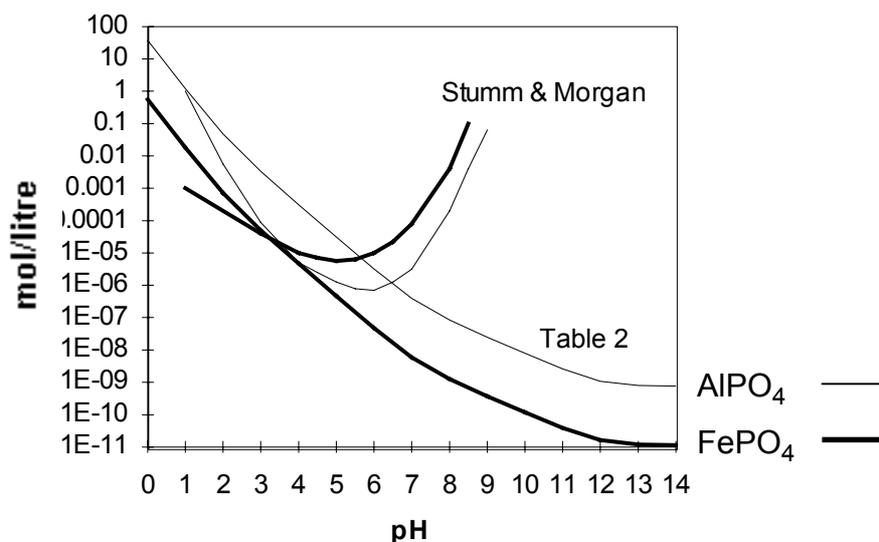
Figure 2. Equilibrium concentration for FePO$_4$ and AlPO$_4$ in mol/litre in solution versus pH according to table 2 and with regard to hydroxide precipitation (Stumm and Morgan, 1981).

PHOSPHORUS RECOVERY EXPERIMENT

Phosphate leaching from ash

Investigations have been made on phosphorus recovery from incineration ashes (Levlin et al., 1998 and Schmidt, 1998). Three types of ashes was used; ash from digested sludge incinerated in a laboratory oven, bottom ash from Högdalen incineration plant there municipal solid wastes were mixed with 10 % heat dried sludge and fly ash from Igelsta incineration plant where biofuel were mixed with 20 % dewatered sludge. The phosphorus is higher in sludge than in municipal waste or biofuel. Phosphorus in ash from incineration of municipal solid wastes varies from 0.45 % (Bäverman, 1997) to 1.3 % (Kida et al., 1969) and in ash from incineration of biofuel from 0.50 % (Bäverman, 1997) to 1.4 % (Sander and Andrén, 1997). The phophorus content was 2.4 % in Högdalen bottom ash and 2.8 % in Igelsta fly ash. The phosphorus content in ash from incineration of sludge is about 8.3 % (Haglund et al., 1997). The phosphorus content in solid waste ash from Högdalen is 0.44 % and in biofuel ash from Igelsta it is 0.45 %. Since the inorganic content is 2.2 % of dry solids in the biofuel and 42.7 % in the sludge, about 80 % of the ash from the co-incineration originates from the 20 % sludge part.

Figure 3 shows the percentage leached phosphorus of total phosphorus from sludge incinerated in a laboratory oven at 550 ºC, 700 ºC, 850 ºC and 1000 ºC, Högdalen bottom ash and Igelsta fly ash. The acid, 0.5 M HCl, was exchanged every second hour up to a total contact time of 8 hours. Högdalen and Igelsta ash were also used in studies of dissolution of phosphorus at different acidity (Schmidt, 1998). Figure 4 shows percentage remaining phosphorus in ash after leaching and dissolved phosphorus then leaching with liquids of 0, 0.5, 1, 2 and 4 M HCl and a contact time of 4 hours.
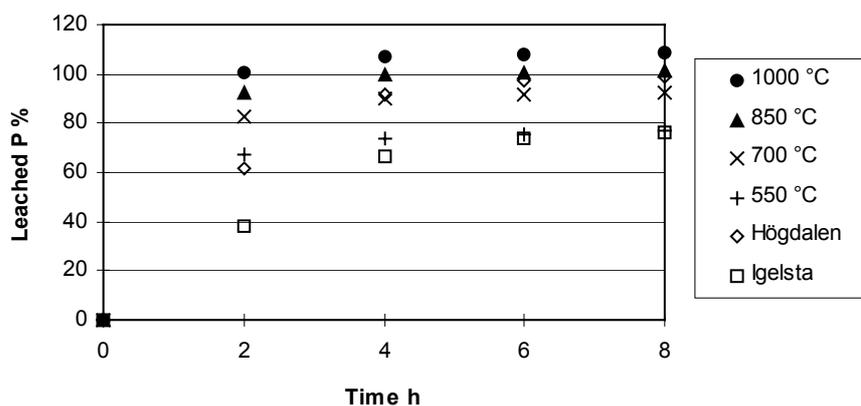
Figure 3. Percentage of phosphorus leached during 8 hours with 0.5 M HCl from incinerated sludge, Högdalen bottom ash and Igelsta fly ash.
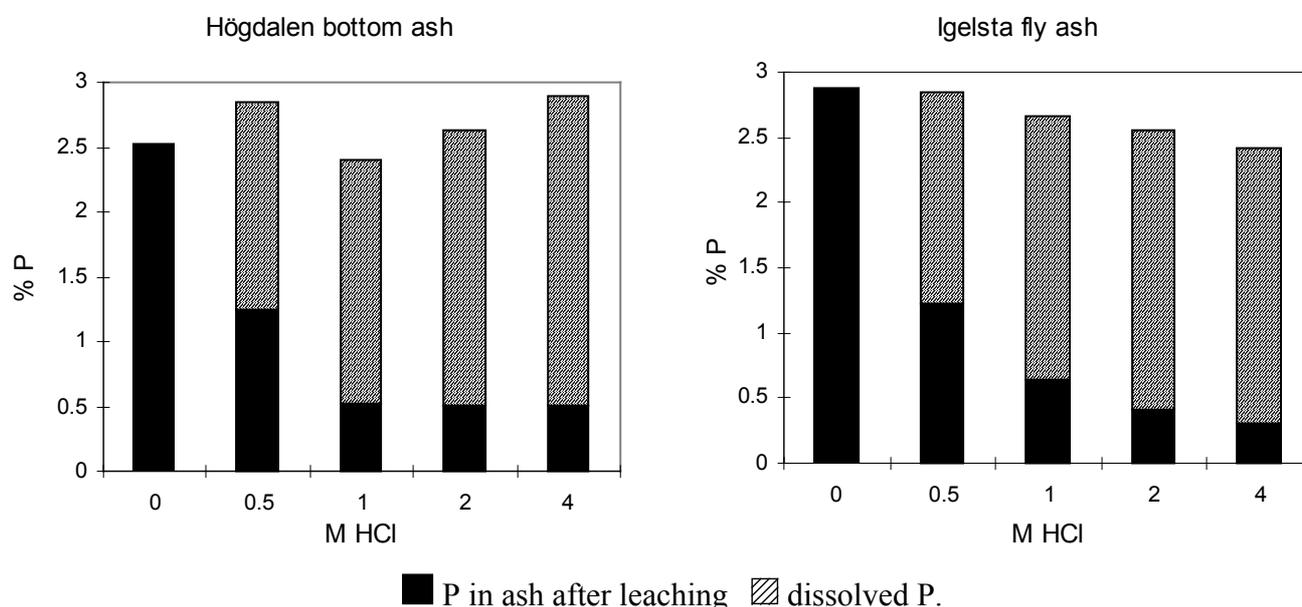
Figure 4. Percentage phosphorus in ash from Högdalen and Igelsta after leaching and dissolved phosphorus versus acidity in M HCl of the leaching liquid.

The figure shows that no acid phosphorus was leached from the ash without acid. However, phsophorus has been leached with warm water from ash of incinerated sludge from biological phosphorus removal in a laboratory study by Matsuo (1996). He also found that addition of iron before incineration decreased the leachability. Use of biological phosphorus removal instead of precipitation with iron sulphate should reduce the inorganic content and thus the ash volumes, increase the phsophorus content in the ash and make the phsophorus more soluble and thus more easy to recover by leaching.

Metal contamination of leachate

Leaching with acid dissolves phosphorus as well as metals, thus contaminating the recovered phosphate with metals. Figure 5 shows that dissolved metal from Högdalen and Igelsta ash increases with the acidity of the leaching liquid.

Högdalen bottom ash

Igelsta fly ash
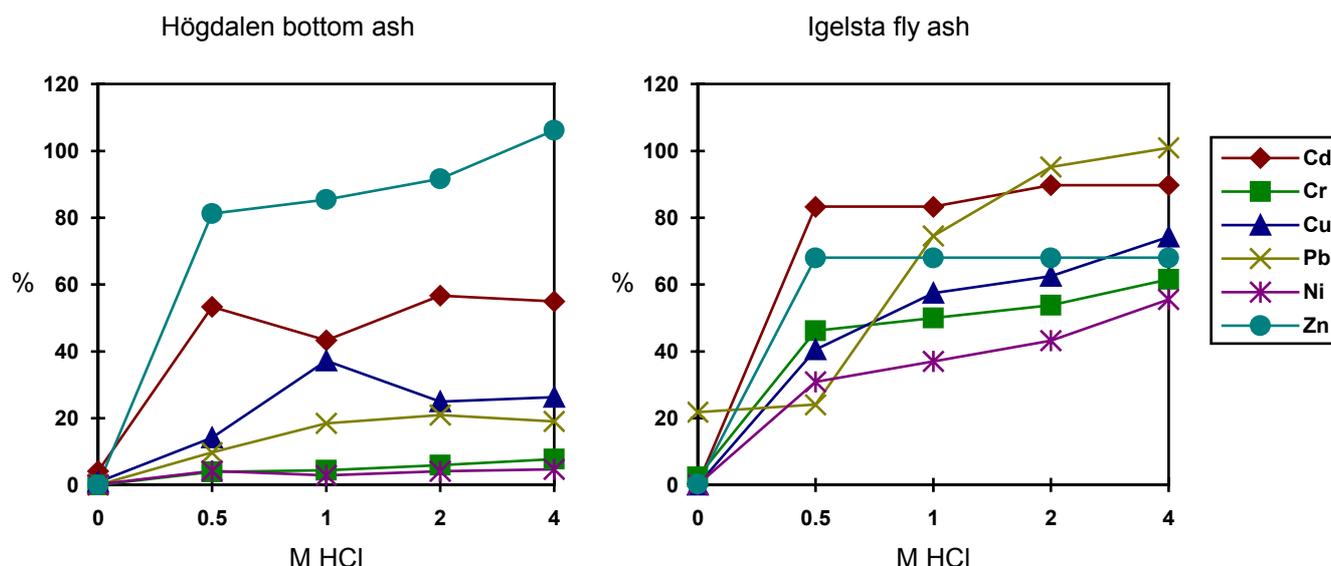
Figure 5. Percentage dissolved metal on leaching Högdalen and Igelsta ash depending on liquid acidity.

When fertilising with recovered phosphate it is the quotient of metal to phosphorus that decides the amount of metal transfered to the soil with the fertilizer. Table 3 shows the ratio metal/phosphorus as mg/g P in sludge, ashes and leachate from leaching with 2 M HCl for the co-incinerations in Högdalen and Igelsta. The quotient for maximum metal content in sludge approved for agricultural use in Sweden for a phosphorus content of 3 % is also shown in the table and quotients higher than in approved sludge are marked bold. Most quotients in ashes and leachate are higher than for the sludge and also higher than in approved sludge.

Table 3. The ratio metal/phosphorus as mg/g P in sludge (3.0 % P), ashes from test incineration in Högdalen (2.42 % P) and Igelsta (2.79 % P), leachate (with 2 M HCl) from Högdalen and Igelsta ashes and for maximum metal content in sludge approved for agricultural use.

| | | Högdalen (10% sludge + 90% waste) | | | Igelsta (20% sludge + 80% biofuel) | | | Sludge approved |
		Sludge, mg Me/g P	Ash, mg Me/g P	Leachate, mg Me/g P	Sludge, mg Me/g P	Ash, mg Me/g P	Leachate, mg Me/g P	for agriculture, mg Me/g P
Cadmium	Cd	0.0533	**<0.12**	**0.08**	0.06	**1.32**	**1.63**	0.0667
Chromium	Cr	**3.67**	**17.4**	1.17	1.13	**22.03**	**16.3**	3.33
Copper	Cu	13.3	**152.9**	**43.4**	13	**501.7**	**43.1**	20
Lead	Pb	1.13	**41.3**	**9.86**	1.53	**73.9**	**96.9**	3.33
Mercury	Hg	0.051	<0.002	---	0.08	**0.16**	---	0.0833
Nickel	Ni	0.8	**19.0**	0.904	0.77	**2.74**	1.63	1.67
Zinc	Zn	22.67	**49.6**	**51.6**	19.67	**498.3**	**466.7**	26.7

Table 3 shows that it is better to fertilize with sludge than with phosphate recovered from ash. It is only the chromium content in the sludge incinerated in Högdalen that is higher than for approved sludge. However, this can be an effect of the large part of waste and biofuel used in the co-incineration. The sludge has about six times higher phosphorus content than solid waste and biofuel, but the metal content is almost the same (see Table 4). Phosphorus from the sludge is thus contaminated with metals from the solid waste and biofuel which makes the metal/phosphorus ratio to be higher in the ash than in the sludge.

Table 4. Metal content in sludge, waste, biofuel and ash for test incineration in Högdalen (Älvesand, 1998) and Igelsta (Haglund, 1998).

		Högdalen (10% sludge + 90% waste)			Igelsta (20% sludge + 80% biofuel)		
		Sludge mg/kg DS	Waste mg/kg DS	Bottom ash mg/kg DS	Sludge mg/kg DS	Biofuel mg/kg DS	Fly ash mg/kg DS
Cadmium	Cd	1.6	5.7	<3	1.8	0.5	39
Chromium	Cr	110	20	420	34	32.6	650
Copper	Cu	400	460	3700	390	64.6	1480
Lead	Pb	34	270	1000	46	33.3	2180
Mercury	Hg	1.53	1.1	<0.05	2.4	0.1	4.4
Nickel	Ni	24	12.6	460	23	2.8	81
Zinc	Zn	680	780	1200	590	636	14700

METAL VOLATILITY

Metals can be removed from the ash if the metals are evaportated at incineration and phosphorus is recovered from the bottom ash. This can be shown by the very low mercury quotient in Högdalen bottom ash. Figure 6 shows the quotient of metal content before and after incineration for Högdalen bottom ash from and Igelsta fly ash versus metal boiling temperature (Levlin et al., 1998). The figure shows that cadmium and mercury is evaporated from bottom ash.
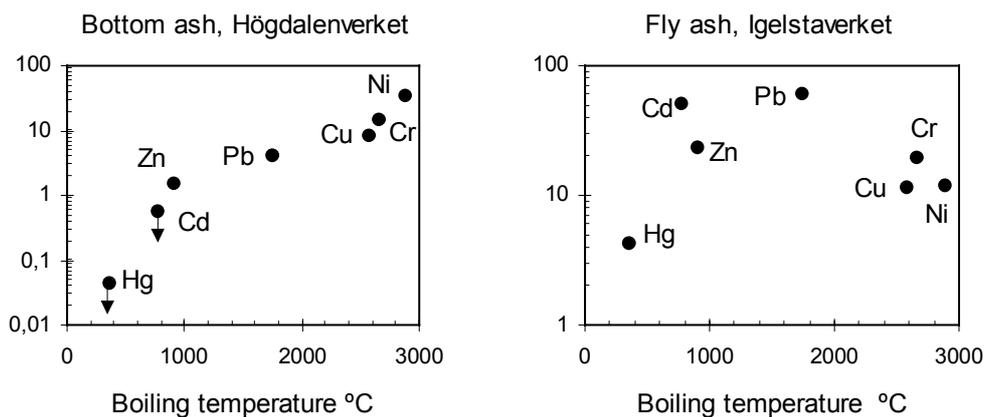
Figure 6. The ratio of the metal content before and after incineration versus boiling temperature for Pb 1743 ºC, Cd 767 ºC, Cr 2663 ºC, Cu 2573 ºC, Hg 357 ºC, Ni 2887 ºC and Zn 911 ºC, for Högdalen bottom ash and Igelsta fly ash (Levlin et al., 1998).

However, the volatility of the metal depends on the boiling temperature as well as on the volatility of metal compounds. For instance chromium may at combustion be oxidized to Cr_2O_3, which has a high boiling point (3000 ºC), or converted to chromyl chloride, CrO_2Cl_2, which has a low boiling point (116 ºC) (Nriagu and Nieboer, 1988). However, studies of formation of chromium compounds at temperatures from 650 ºC to 1050 ºC shows that the only chromium product of significans is Cr_2O_3 and that chromium (VI) is less than 10% of the total chromium product. If nickel is present $NiCr_2O_4$ is produced. Calculations by Wendt and Linak (Tillman, 1994) shows that, at thermodynamic equilibrium, chromium is influenced by chlorine, particulary at low temperatures. Further, this influence is moderated by sulfur (see figure 7). Chlorine may increase the volatility especially around 500 ºC. Chromium in the solid phase is trivalent Cr_2O_3 and in the gas hexavalent CrO_2Cl_2. At higher temperatures chlorine is less significant as an oxidant, and oxygen is more significant. Under these conditions chromium remains as a refractory metal and is at higher

temperatures as trivalent Cr_2O_3. Combustion occurs at higher temperatures than 800 ºC, there volatility of chromium is lower. Thus, a possibility to remove chromium is to add salt and treat with heat at 500 ºC, either before or after incineration.
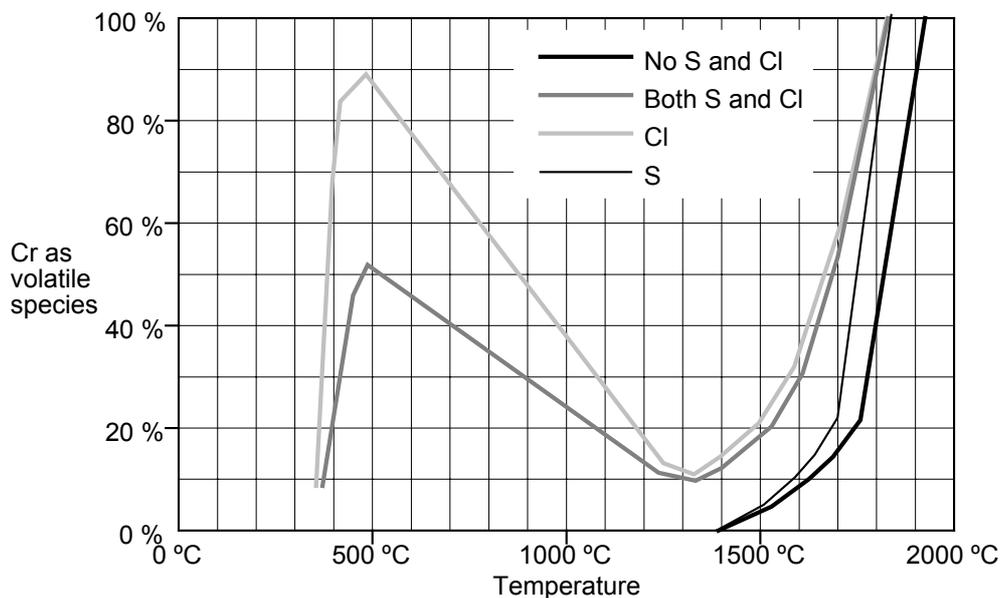
Figure 7. Influence of temperature, Cl and S on the volatility of Cr (Tillman, 1994).

Chromyl chloride, CrO_2Cl_2, is a liquid at room temperature with a boiling point at 116 ºC (Nriagu and Nieboer, 1988). It is a chromium (VI) compound used extensively in organic synthesis. It hydrolysis in contact with water, yielding H_2CrO_4 or its dissociation products, $H_2CrO_4^-$ and CrO_4^{2-}.

$$CrO_2Cl_2(l) + 2H_2O(l) \rightarrow H_2CrO_4(aq) + 2HCl(g)$$
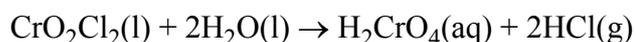
This reaction is exothermic and explains the fuming of CrO_2Cl_2 in moist air. Chromyl may form compounds with other halides (especially fluoride) or other anions (nitrate, cyanate or acetate). These chromyl compounds, like the chloride derivate, are volatile materials, although there are wide differences in their boiling points and volatilities. Chromyl chloride gas can at 360 ºC be converted to CrO_2 and Cl_2 (Gmelins Handbuch der Inorganische Chemie 1962).

UTILISATION OF ASH FOR BUILDING MATERIAL

To avoid deposition, the inorganic material remaining after incineration can be used for production of building materials. Any environmental hazardous contaminants are bound as mineral to the material and utilisation of ashes reduces the need for mining of raw material for production of building material. The ash can be used for production of building materials in three ways;. the ash can be mixed with cement or concrete, a brick or some other object can be made of ash and the ash can be melted and solidified as a ceramic material.

For production of bricks and cement the sludge can be used without incineration. The high aluminium content of a sludge produced at water treatment with aluminium salts makes it useful for production of aluminous cement (Lubarski et al., 1996). The evaporation of the volatile part of the sludge makes bricks produced of sludge and clay porous. The solubility and toxicity of chromium is higher than in the sludge,

especially if the sludge is used for brick manufacture there alkaline components are added before incineration (Rump, 1998). Hexavalent chromium (VI) is carcinogenic and it is often regulated as if all of the chromium resulting from combustion processes is in the hexavalent oxidation state (Tillman, 1994). The redox potential of the sludge is low due to microbial oxidation of the biological material. Due to increasing redox potential, incineration increases the solubility of chromium, since insoluble chromium (III) is oxidised into hexavalent (VI) soluble form.

Bricks can be made from ash (Daido, 1998) by charging ash into a metallic mold with the shape of the wanted brick and pressed into semi-manufactured bricks. The pressed bricks are than sintered in a kiln type furnace at 1000 ºC during 10 - 12 hour. One problem with the bricks is the higher water adsorption caused by capillary action of many fine pores. To decrease the water adsorption the bricks were soaked into a silocone solution. Another problem is the variation of the used ash. Since the components of ash are changeable mainly by the influence of rain, the receiving of ash was stopped on rainy days. Further, the ash components were analyzed with fluorescence X-ray method and an equation was introduced for calculating the suitable sintering temperature.

Making of brick by mixing ash with clay has been studied by Grehl and Müller (1998). They recomended that the maximum ash content should be 25 % of dry solids.

A ceramic material can be produced by melting the ash and which thereafter is solidified (Nomura, 1998, Endo et al., 1997). A high content of SiO_2 and Al_2O_3 and a low CaO content makes the melted ash to crystallise as mullite in the phase diagram SiO_2 - Al_2O_3 - CaO shown by Figure 8. However, crystallisation as anorthite will give the glass ceramic better material properties, which can be achieved by using CaO as sludge conditioning agent instead of polymers.
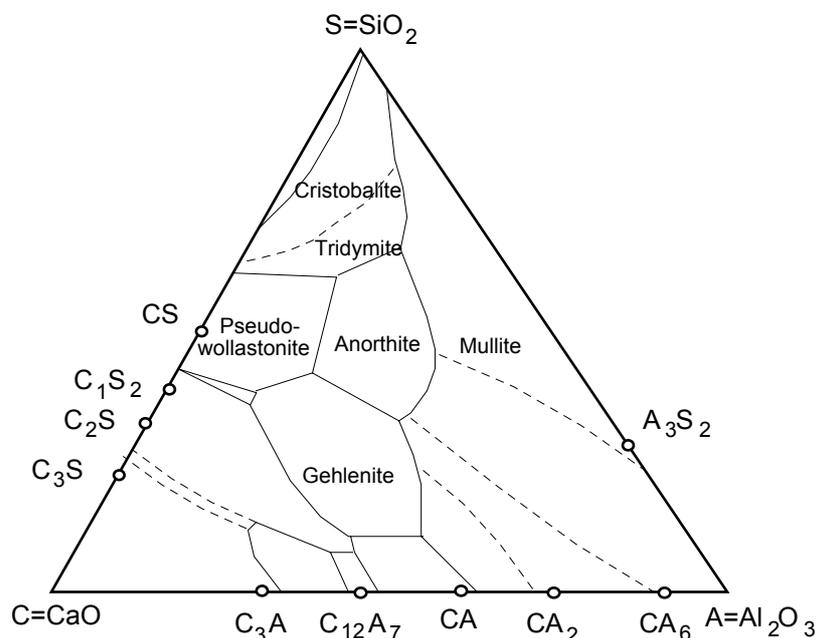
Figure 8. Phase diagram for the system SiO_2 - Al_2O_3 - CaO.

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