



## Potential of thermal treatment for decontamination of mercury containing wastes from chlor-alkali industry

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### ABSTRACT

Old dumps of mercury waste sludges from chlor-alkaline industry are an environmental threat if not properly secured. Thermal retortion can be used to remove mercury from such wastes. This treatment reduces the total mercury content, and also may reduce the leachability of the residual mercury. The effects of treatment temperature and treatment time on both residual mercury levels and mercury leachability according to the US EPA TCLP leaching procedure, were investigated. Treatment for 1 h at 800 °C allowed to quantitatively remove the mercury. Treatment at 400 °C and above allowed to decrease the leachable Hg contents to below the US EPA regulations. The ultimate choice of treatment conditions will depend on requirements of further handling options and cost considerations.

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### 1. Introduction

High mercury-containing wastes can constitute a significant environmental threat when not handled properly. The amount of Hg in wastes only in the EU has been estimated at about 990 metric tonnes [1]. An important industrial source of high mercury waste is the electrochemical production of chlor-alkali from cells with mercury cathodes [2–5]. Although at present membrane cell and diaphragm cell processes are replacing Hg cell technology, there are hundreds of tons of Hg contaminated wastes accumulated and buried. This research concerns mercury waste disposed in the central region of Cuba Island. The current storage of the waste in concrete niches is inadequate, allowing Hg to leach from the waste. The regular occurrence of natural disasters such as hurricanes, inundations and earthquakes aggravates the risks for dispersion of Hg into the environment.

Recycling of waste is higher up the "waste management hierarchy" than landfill disposal [1]. If economically or environmentally viable, hazardous waste should be recycled, limiting the risk to environment and public health. Technologies applied for removal or stabilization of Hg in contaminated solid waste or soil include solidification/stabilization [2,6–10], soil washing [10–12], thermal

treatment [10,13–16], and vitrification [10]. A comparison of the different technologies is provided in [10]. Retorting is a thermal treatment method involving distillation or dry distillation [13]. It is a method recommended by US EPA to treat high mercury content waste (>260 mg/kg) [17].

The present communication aims to investigate the effects of temperature and exposure time on the efficiency of mercury reduction of waste sludge from the electrochemical process of chlor-alkaline production. The effect of the treatment is evaluated based on reduction in total contents and reduction in leachability according to the US-EPA Toxicity Characteristic Leaching Procedure [18].

### 2. Experimental

#### 2.1. Mercury waste sampling and preparation

The solid waste samples used for this study were collected from two different niches. In each niche, samples were collected at three points on a diagonal line, in the middle and at 20 cm of the two extremes, and at each point from three depths (0–20, 20–40 and 40–60 cm). These samples were combined in order to obtain two composite samples representative for each niche. In the laboratory, sludge samples were air-dried for seven days. They were hand-crushed in a mortar, passed through a 2-mm sieve and thoroughly mixed. The samples are referred to as Sample A and Sample B.

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## 2.2. Sample characterization

Cation exchange capacity (CEC) at neutral pH was determined by the ammonium acetate method using 5 g of mercurial waste [19]. To determine the chloride content 1 g of sediment was suspended in 50 ml of 0.15 mol/l  $\text{HNO}_3$  and shaken for 30 min. The filtrate was titrated with 0.05 mol/l  $\text{AgNO}_3$  using potentiometric end-point detection [19] by potentiometer Metrohm, 761 Compact IC, Switzerland. Organic matter was estimated through loss on ignition (LOI) method as is reported by Heiri et al. [20] for organic matter determinations in soils. In this test 5 g of the sludge sample were heating during 1 h at 400 °C and the weigh loss was determined and assumed as represents the organic matter content in the sludge. The metals content in mud samples (pseudo-total content) was determined using 1 g of the sludge by digestion in aqua regia [21] for 2 h at 150 °C of temperature, employing the Inductivity Coupled Plasma with Optical Emission Spectrometry (ICP-OES, Vista MPX CCD Simultaneous, Varian, Australia) method. All analysis was carried out in triplicate.

## 2.3. Mercury determination

Total mercury content was determined by Cold Vapour Atomic Absorption Spectrometry (Mercury Analyzer MAS-50, Coleman, IL, USA) after a specific destruction, that involved the addition of 0.05 g of  $\text{V}_2\text{O}_5$ , and 10 ml of concentrated  $\text{HNO}_3$  to 0.5 g of sample, and digesting for 30 min at 160 °C [22]. After cooling down, 15 ml of concentrated  $\text{H}_2\text{SO}_4$  was added. The digestion was continued for 2 h at 160 °C. To measure Hg, the digest was transferred to a mercury reduction vessel. After addition of  $\text{SnCl}_2$ , the solution was purged and the gasses were introduced into the mercury analyzer. Peak absorbance was recorded. A standard solution of 0.25 mg/l Hg was prepared from 1000 mg/l stock solution (MERCK, Darmstadt, Germany). The calibration curve was obtained by injecting different volumes of this 0.25 mg/l stock solution (0, 0.5, 1, 2, 4 and 8 ml) into the Hg reduction vessel. The calibration was used only when a correlation better than 0.99 was achieved. All reagents used were analytical grade.

## 2.4. TCLP tests

Mercury toxicity levels in the mercury waste and the ashes resulting from the thermal treatments were evaluated according to the EPA TCLP Procedure [23]. The extraction fluid used depends on the alkalinity of the residual solid phase. According to the TCLP procedure, pH (1) value was measured by mixing 5 g of sludge sample and 96.5 ml of deionized water in a 250 ml beaker after stirring for 5 min. This pH-value provides an indication of the acidic or alkaline properties of the waste. The value of pH (2) is tested after adding 3.5 ml 1 mol/l HCl to the suspension, followed by heating to 50 °C for 10 min. This provides an indication on the alkalinity of the waste solids. If pH (2) is below 5, an acetic acid/acetate buffer solution is used (extraction fluid #1). Otherwise, a more acidic extractant fluid #2, composed of 5.7 ml glacial  $\text{CH}_3\text{CH}_2\text{OOH}$  in 1 l deionized water, must be used. Both samples required the use of the latter extracting solution.

For extraction, 1 g of the sample was introduced in 100 ml high density polyethylene (HDPE) extraction vessels, and 20 ml of extractant fluid 2 was added. Suspensions were agitated during 18 h in the end over end shaker operating at approx. 30 rpm. After agitation, the final pH, pH (3), was measured. The TCLP leachates were filtered using 0.45  $\mu\text{m}$  pore membrane filters (CM, Porafil, Macherey-Nagel, Düren, Germany). The filtrates were acidified to pH < 2 using concentrated  $\text{HNO}_3$  and stored at 4 °C before analysis. Analysis of Hg determination was carried out by CVAAS as described before.

## 2.5. Thermal treatment

Experiments at different temperatures and times were carried out at the laboratory scale to simulate the retorting conditions. A muffle furnace (Carbolite type P330 Controller, Nabertherm, Bremen, Germany) located inside a fume cupboard Model Potteau, Belgium, was used. Each test involved ashing 5 g of dried and ground mercury waste in a porcelain crucible in different conditions. In a preliminary test series, mercury removal after treatment at 600 and 800 °C during 30 and 60 min was evaluated. No replicates were done. In a second test series, temperature was varied between 100 and 800 °C in increments of 100 °C. Retorting time was 1 h. These tests were triplicated.

## 3. Results and discussion

### 3.1. Chemical and physical characterization of samples

The wastes reveal themselves as grey powders. The pH in water is high, at about 10. This would suggest the presence of lime compounds ( $\text{CaO}$ ,  $\text{MgO}$ ) next to lime stone ( $\text{CaCO}_3$ ,  $\text{MgCO}_3$ ). Compounds where limestone dominates would exhibit a pH between 7.5 and 8.5 [24]. No organic carbon determination is available, but for soils and sediments, loss on ignition at 400 °C can provide a good estimate of organic matter [20]. Although this estimate is less precise for waste materials, weight loss of the waste at 400 °C may indicate organic matter levels in the order of 8–10%. The wastes exhibit some cation exchange capacity, although it is small and comparable to typical light textured soils [25]. As such, it is not expected to be capable of retaining significant amounts of cations on the sorption complex. Differences in CEC between the samples could be related mostly to differences in organic matter content, which is a prime component contributing CEC to a soil [25].

The mercury wastes contained high levels of Ca, Mg, and Na (Table 1). The presence of Na is associated with the brine used for electrolysis, the use of  $\text{Na}_2\text{CO}_3$  and NaOH used for the purification of the brine, and the use of NaS for precipitation of Hg. The presence of Ca and Mg is explained by to the use of calcareous products such as  $\text{CaCO}_3$  and  $\text{Mg}(\text{OH})_2$  to increase pH and stabilize the sludge. Significant amounts of  $\text{Na}^+$  are explained by the use of NaCl in the electrolytic process and the high Fe values are provided by diatomaceous earth composition, which also contributes to stabilization

**Table 1**

Properties and metal contents of mercury sludge samples (mean  $\pm$  standard deviation,  $n = 3$ ).

Parameter	Sample A	Sample B
pH-H <sub>2</sub> O	9.96 (0.01)	9.98 (0.02)
Chloride (g/kg)	178 (1)	94 (2)
Org Matter Content (g/kg)	85(9)	100 (8)
CEC (cmol <sub>c</sub> /kg)	3.2 (0.3)	6.7 (0.4)
Total Metal Content (g/kg DM)		
Ca	156 (5)	125 (1)
Mg	46.5 (0.1)	36.3 (0.3)
Na	128 (3)	36(0.7)
Fe	4.97 (0.05)	4.93 (0.03)
K	1.01 (0.001)	0.54 (0.003)
Total Metal Content (mg/kg DM)		
Cd	1.06 (0.02)	1.99 (0.01)
Cr	15.0 (0.6)	19.0 (1.3)
Cu	63 (3)	170 (2)
Mn	132 (1)	136(0.1)
Ni	10.4 (0.4)	10.6(0.3)
Pb	15.1 (0.06)	26.9 (0.6)
Zn	159 (2)	121 (2)
Hg	505 (17)	1205 (16)

**Table 2**

pH and Hg concentrations in extracts according to the TCLP leaching test (mean  $\pm$  standard deviation,  $n=3$ ).

Sample	A	B
pH (3)	7.84 (0.16)	8.07 (0.07)
Hg (mg/l)	3.85 (0.24)	0.32 (0.05)

Mercury content by EPA Regulations: 0.20 mg/l.

of the wastes. The typical chemical composition of diatomaceous earth includes about 2% iron [26].

As expected, the waste materials contain significant amounts of Hg, at 505 mg/kg dry matter for the first sample and 1205 mg/kg for the second. Considering levels that naturally occur in soils (<0.4 mg/kg as a world wide average in soils as is reported by Kabata-Pendias and Pendias [27]), these concentrations are extremely high. With Hg total contents exceeding 260 mg/kg, the waste is characterized as high mercury waste according to the US Land Disposal Restrictions. Important differences in Hg contents and other properties between the samples reflect the two different niches, where sediment from different batches and time periods were disposed. Unfortunately, there is no record on the exact time of disposal in these niches.

Other potential toxic trace elements were analysed to assess whether they could also be of environmental concern. Chromium, Mn, Ni, and Pb were well within levels normally present in soils [25], and thus are not expected to pose any problem. Contents of Cd, Cu and Zn were somewhat elevated compared to normal ranges in soils, but still in the same order. Hence, no significant issues are anticipated also with these elements, although a final conclusion would require further testing.

Mercury leaching from both samples exceeded the EPA TCLP limit of 0.2 mg/l (Table 2). Hence, the waste is classified as toxic waste according to US EPA regulations. The significant difference in leachability between samples, an order of 10, reflects the different nature of the waste in the various niches, and the variability in composition and behaviour of wastes generated at different times. Surprisingly, the waste with the higher total mercury content (waste B) is the one with the least Hg leaching. This could partly be explained by a higher buffering capacity, which is reflected in a higher pH after the extraction. Theoretically,  $\text{Hg}(\text{OH})_2$  solubility is expected to decrease by a factor 100 for each unit increase in pH [24]. As such, a pH difference of only 0.25 units is very important with respect to leaching behaviour.

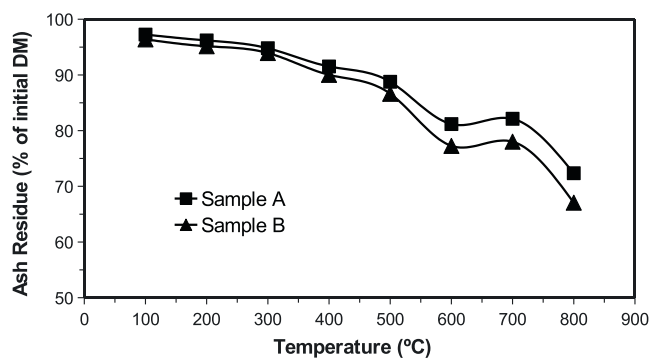
### 3.2. Thermal treatment

Table 3 shows ash rest and mercury removal efficiency at 600 °C and 800 °C, for 30 min and 1 h. Efficiency was calculated as the difference between mercury content in the sludge sample before and after the treatment divided by the mercury content before the retorting treatment. The lowest values of ash content and average Hg content in the remaining ash were consistently obtained at higher temperature and exposition time. The effect of temperature was much stronger than the effect of time. In a follow up experiment, the exposure time of 1 h was

**Table 3**

Effects of time and temperature on residual ash content, residual Hg content and Hg removal efficiency.

Temp. (°C)	Time (min)	% ash		Residual Hg (mg/kg)		Treatment efficiency (%)	
		A	B	A	B	A	B
600	30	86.4	81.4	12.4	11.8	97.6	99.0
800	30	74.0	68.7	1.25	0.52	99.7	100.0
600	60	85.0	80.1	9.9	8.6	98.0	99.3
800	60	71.9	65.9	0.18	0.11	100.0	100.0



**Fig. 1.** Influence of retorting temperature during 1 h on the residual ash, expressed as percentage of the initial dry mercury sludge.

**Table 4**

Total mercury and mercury removal efficiency remaining after thermal treatment of the mercury sludges as a function of treatment temperature.

Temp. (°C)	Residual Hg content (mg/kg on ash) <sup>a</sup>		Hg removal efficiency (%)	
	A	B	A	B
20	505 (17)	1205 (16)	–	–
100	489 (23)	1473 (190)	3.1	–22.3
200	236 (34)	814 (34)	53.3	32.5
300	40 (3)	47 (6)	92.1	96.1
400	31 (3)	33 (3)	93.8	97.3
500	20.0 (0.7)	20.0 (0.8)	96.1	98.4
600	10.1 (0.9)	11.2 (2.1)	98.0	99.1
700	3.1 (1.2)	2.1 (0.9)	99.4	99.8
800	0.10 (0.07)	0.10 (0.01)	100.0	100.0

<sup>a</sup> Average and standard deviation of 3 replicates.

adopted, and the effect of temperature was investigated in more detail.

Fig. 1 reveals the decrease in ashes residue with temperature. Although weight loss of both samples followed the same trend, sample A exhibited less decrease in weight than sample B. Organic matter content and loss of crystalline bound water mostly explain weight decreases up to 400–500 °C. Above that temperature, carbonate compounds increasingly will decompose to oxides, e.g. CaO and MgO [20]. This is observed by the additional decrease in ash residue above 500 °C.

Up to 300 °C the removal of Hg was low, below 50% (Table 4). Similar results were obtained by Taube et al. [3] but working with mercury contaminated soils. Mercury removal became highly efficient (more than 90% removal) when temperature was higher than 300 °C. Similar results were reported by Chang and Yen [16]. Samples behave with differences between them at low temperatures (<400 °C) and in similar way above 600 °C.

### 3.3. Leachability of the treated wastes

Fig. 2 shows the final pH after extraction as a function of temperature. Considering that the initial pH of the extracting solution is 2.88, the high pH values after extraction reveal the large

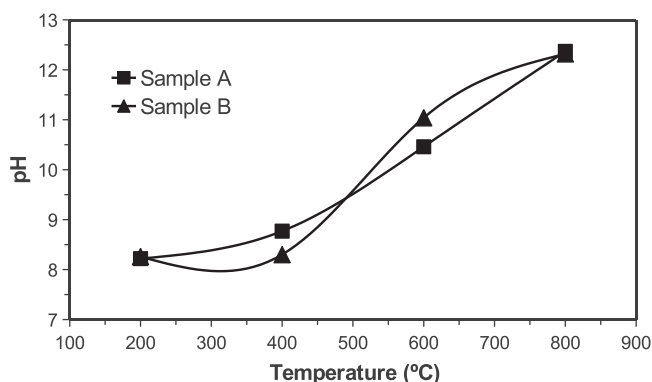


Fig. 2. Final pH of the TCLP extract of mercury sludges, treated during 1 h at increasing temperatures.

buffering capacity of the wastes. The extraction fluid used consisted of 0.1 mol/l acetic acid. In the conditions of the extraction, at most 0.1 g of  $\text{CaCO}_3$ -equivalent can be neutralized. The excess of calcareous materials therefore determines the pH after extraction. The increase in final extraction pH above 500 °C reflects the increasing conversion of carbonates to oxides during the retorting process. Whereas carbonate compounds governed the final pH under 500 °C, increasingly hydroxides governed the final pH at increasing temperatures. Fig. 3 reveals a decrease in TCLP leachable Hg with temperature. Treatments at temperatures above about 350 °C allowed to decrease Hg leachability in the residue to below the TCLP reference value, and would cause the product not anymore to be classified as toxic waste. Current experiments reveal that technically, the thermal treatment allows decreasing total mercury contents, and consequently also achieves to reduce leachability to values below the threshold value.

The optimal operating conditions for the retorting operations will depend on subsequent handling options for the treated waste. Provided total contents of other elements are not a limiting factor, the material may be considered for reuse in construction or landscaping. This would require retorting conditions that reduce total mercury contents to below limits considered acceptable for this type of use. In case the treated material is to be disposed again, milder conditions for retorting could be used, that decrease mercury leaching to below the threshold level, without eliminating all total mercury contents. While the first option would lead to an elimination of this waste problem, associated costs might be prohibitive. Economic considerations in combination with different requirements for different handling options e.g. reuse or disposal, will ultimately determine the optimal treatment conditions of the retortment process.

As an element, mercury is never destroyed in any waste treatment process, but only transferred. The retorting process, which is

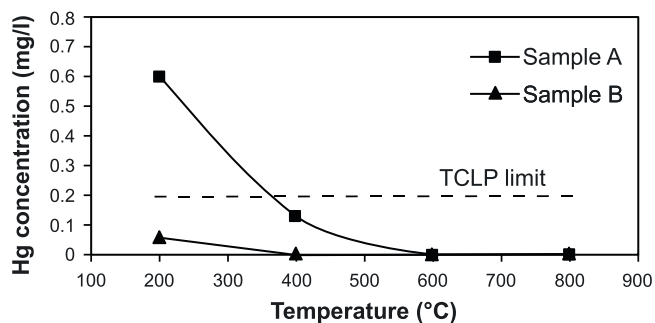


Fig. 3. Mercury concentrations in TCLP extracts of mercury sludges, treated at increasing temperatures.

based on volatilisation and subsequent condensation of the mercury vapour, results in the recovery of metallic mercury [13,16]. As such, 1000 m<sup>3</sup> of mercury sludge would yield about 0.125 m<sup>3</sup> of metallic Hg, assuming a bulk density of 1700 kg/m<sup>3</sup> for the sludge. The recovered metallic mercury has a purity of 99% [16] and could be recycled or be disposed in smaller containers depending of the industrial management interest. These aspects require further investigation, preferably based on pilot scale studies.

#### 4. Conclusions

The waste sludge of chlor-alkali industry that was studied is considered as a toxic waste, based both on total contents and leachability of Hg. The current storage in poorly confined niches constitutes a significant risk for Hg dispersion into the surrounding environment. Thermal treatment studied is very effective to remove the Hg, considering the high mercury removal efficiency, close to 100% obtained in this study. Leachability according to the US EPA TCLP leaching test decreases below the threshold value of 0.2 mg Hg/l after treatment at a temperature of 400 °C or higher. Optimal operating conditions will depend on the requirements of further handling options and cost considerations.

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