

Review

Thermal treatment of hazardous waste for heavy metal recovery

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Abstract

In this study, a new method for recovering heavy metals from hazardous waste is introduced. The process is characterized by a separation of heavy metals and residues during the thermal treatment under a sub-stoichiometric atmosphere in a rotary kiln. After leaving the rotary kiln the separated heavy metals are precipitated in a hot gas ceramic filter. Using this technology, hazardous materials, both liquids and pasty hazardous waste containing heavy metals, can be treated and a product with a quasi-raw material condition can be formed. In contrast to current methods, the harmful substances should not be immobilized and disposed. In fact, a saleable product highly concentrated with heavy metals should be formed. During preliminary investigations with a solution containing sodium chromate tetrahydrate, the process was tested in a pilot plant. Here, the separation of chromium could be demonstrated with leaching tests and characterization of the filter dust. Analysis concerning the disposability of the residues had not been carried out because only the process and the characteristic of the filter dust were in the centre of attention.

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1. Introduction

According to [1], the volume of waste accumulating in Germany looks set to decline. However, although this may be true of the total volume of waste, further examination reveals that this overall decrease will mainly result from the amount of build-

ing and demolition waste shrinking. By contrast, the volumes of waste from production and industry are likely to remain more or less unchanged. These trends are outlined in Fig. 1.

Moreover, as far as the percentage of hazardous waste is concerned (cf. Fig. 2), an increase is apparent over the period of investigation. The proportion of waste materials from production and industry for instance rose from 16.1% in 1999 to 20.5% in 2003. The reason for this, however, is not the failure of the avoidance principle as enshrined in Section 3 KrW-/AbfG (Germany Recycling and Waste Act), but is instead attributable to repeated

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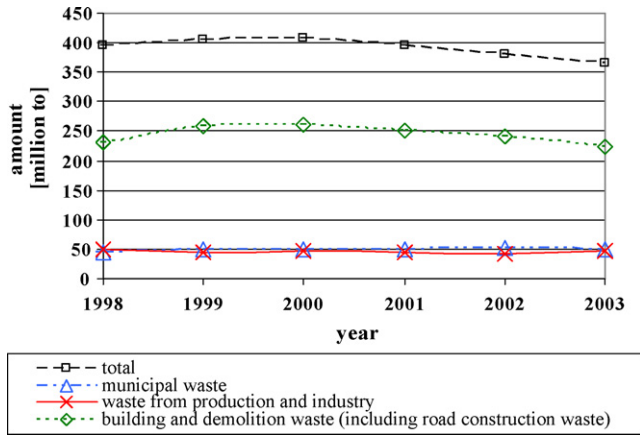


Fig. 1. Development of the amount of waste according to [1].

changes to the legal basis for the assessment of hazardous waste.

Until 31 December 1998 the classification of waste materials was carried out depending on the materials' properties based on the LAGA (working group for waste topics of German states) catalogue of waste materials. Since 1 January 1999 waste materials have been categorized by their origin by applying the waste catalogue according to the Ordinance to Implement the European Waste Catalogue (EWC).

In particular, mixtures of building waste comprising mainly glass, wood and plastic materials have now been declared hazardous after having been categorized until 31 December 1998 as not requiring special monitoring.

The most recent change regarding waste requiring special monitoring occurred when the Ordinance on the Waste Catalogue came into force on 1 January 2002, under which waste materials are now also categorized by origin.

Accordingly, the described development of the regulations for categorizing hazardous waste allows only for a conditional interpretation of the volume of waste. It is, however, noticeable that particularly in the field of production and industrial waste (other than building waste), a decrease in the waste volume can hardly be expected. This assumption is underlined by a prediction made in the Berlin waste management blueprint for the

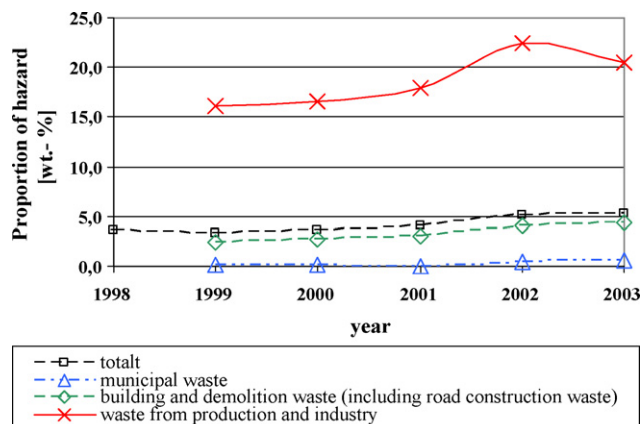


Fig. 2. Development of the proportion of hazardous waste according to [1].

year 2002 (cf. Table 1), which predicted that the current volume of hazardous waste from industry, public institutions and the domestic sector of 149,300 Mg is likely to decline by merely 4.3 wt% by 2015.

According to [3], also the hazardous waste generation in Finland between 1992 and 2003 has increased.

It can therefore be concluded that despite improving waste avoidance and recycling in production, new and above all efficient ways of disposing of hazardous waste such as that described below will still be urgently needed [2].

One of the most hazardous waste streams in metallurgical industry is the galvanic sludge [4]. An often used treatment method for such streams is the incineration with the aim of minimizing environmental risks in the form of toxic substances like heavy metals. The destruction of toxic organic compounds, the immobilization of bio-available inorganic substances and the reduction of the volume are the main advantages of such technologies [5]. Many technologies for hazardous waste thermal treatment are known and discussed. Besides the traditional methods like the incineration, pyrolysis and gasification of hazardous waste, new techniques like the solar treatment [6–8] are in development. In spite of everything, the highest commercial level of operation has reached incineration techniques [9].

In case of a thermal treatment, often the behaviour of heavy metals [10–64] is discussed. The heavy metals should be immobilized and remain in a solid constitution. Reference [9] confirmed this with studies in bubbling fluidised combustor.

By using the following subscribed technique this effect should be connected with the intention of a recycling of heavy metals. In contrast to other treatment methods for hazardous waste, the advantages of the process are

- recovery of heavy metal oxides and oxide mixtures from waste and residue and their transfer into a quasi-raw material condition,
- concentration of heavy metals in the filter dust after separation in the reactor,
- treatment of liquid and past-like hazardous materials,
- generation of a marketable product,
- abandonment of extraction processes [65,66] and following steps like nanofiltration.

The process described in this article is patented worldwide under WO 98/49355 [67] and was published on 5 November 1998. In a project the process's 'functionality' was demonstrated (i.e. its technological and economic suitability for the disposal of heavy metals containing hazardous waste). The process was developed by Dr. Kaszás-Savos and Mr. Kaszás. Other participants in the project were MENTEC GmbH (project organization), Industrieofenbau Aue GmbH (plant engineering), and the Institute of Waste Management at Dresden University of Technology (scientific supervision). The project was kindly co-financed under the European Regional Development Fund's technology funding 2000–2006 and by the government of the Free State of Saxony.

Table 1
Predicted accumulation of hazardous waste in Berlin according to [2]

Waste	Supply in tones			
	2002	2005	2010	2015
Hazardous waste in mineral building waste and excavated soil	585,000	400,000	360,000	300,000
Hazardous waste in industry, the public and domestic sector	145,450	149,300	149,420	148,650
Total	730,450	549,300	509,420	448,650

2. Wastes

The process for the recovery of heavy metal oxides and oxide mixtures is mainly appropriate for residue, sewage and sludge from the following industries:

- surface refinement,
- chemical,
- cosmetics,
- automotive.

Sludge classified under waste numbers 110109 and 110113 as well as wastewater from the surface-refining industry are regarded as potentially suitable for thermal treatment as

described below. This disposal route for sewage contaminated by heavy metals could be a practical option for small businesses in particular, since it obviates the need for very expensive wastewater treatment.

The composition of galvanic sludge for potential application was investigated by the North Rhine-Westphalia State Environment Agency (LUA NRW) from 1990 to 2004 and documented on the Galvanization Sludge Waste Datasheet. The composition was not found to vary significantly regardless of the time frame, branch of industry or disposal route. The composition of galvanic sludges shown in Table 2 is therefore still valid and can serve as a basis for further calculations.

Above all, the broad spectrum of heavy metals (largely due to the metals used during electroplating) is apparent. Particularly

Table 2
Composition of galvanization sludge (sludge from electroplating), modified according to [68]

Parameter	Unit	Number of measurements	Maximum	80% percentile	Median
Solid matter water content	%	315	84	67	60
Loss on ignition	%	485	85	30	21
Calcium carbonate	%	9	18	11	1.8
Anions					
Chloride	mg/kg DM	20	39,200	6,780	115
Cyanide (readily releasable)	mg/kg DM	61	57	1	0.1
Fluoride	mg/kg DM	23	230,000	45,540	62.5
Total phosphate	mg/kg DM	25	140,000	69,800	35,400
Sulfate	mg/kg DM	14	29,000	28,000	23,750
Light metals					
Aluminum	mg/kg DM	30	316,000	240,000	69,100
Barium	mg/kg DM	17	830	586	295
Calcium	mg/kg DM	28	330,000	179,240	54,670
Potassium	mg/kg DM	15	13,600	3,044	300
Magnesium	mg/kg DM	15	13,000	2,220	1,600
Sodium	mg/kg DM	17	22,000	17,980	4,100
Titanium	mg/kg DM	5	1,420	1,132	520
Semi-, heavy-, noble metals					
Antimony	mg/kg DM	40	2,946	1,103	16
Arsenic	mg/kg DM	102	249	17.9	6.2
Lead	mg/kg DM	130	107,000	790	157
Cadmium	mg/kg DM	130	8,500	10.06	1.3
Chromate(VI)	mg/kg DM	15	5	8	0.4
Chromium (total)	mg/kg DM	143	300,000	43,144	13,968
Iron	mg/kg DM	75	580,000	120,000	30,000
Copper	mg/kg DM	145	670,000	21,520	2,100
Nickel	mg/kg DM	150	260,000	45,040	2,805
Zinc	mg/kg DM	146	688,000	65,903	7,080
Cobalt	mg/kg DM	39	517	57	14
Organic parameters					
TOC	%	233	33	6.7	2.4

DM: dry matter.

high concentrations of aluminum, iron, chromium, zinc, nickel and copper were found.

The total chromium content accounted for maximum 30 wt%. In 80% of the sludge analyzed, it was 0.43 wt%. The need to recover this very high potential of chromium in galvanic sludge is obvious.

By contrast, the levels of manganese, tin, lead, antimony, cadmium, silver, mercury and arsenic are lower, while significant concentrations of cyanides, nitrides and fluorides were rarely found.

In 2004, galvanic waste was treated as follows in Germany:

- 68.2 wt% deposited on the surface,
- 10.9 wt% recycled,
- 9.3 wt% physicochemically treated,
- 4.5 wt% disposed of underground,
- 2.6 wt% subject to interim deposition,
- 0.7 wt% thermally treated.

This comparison highlights the very minor role played by thermal treatment (the incineration of hazardous waste) and the high proportion of surface deposition. The proportion of surface and underground disposal is expected to decline over the next few years (because these disposal routes correspond to little more than packaging) in favor of material recovery and high-grade utilization. Once again the relevance of developing and optimizing processes for recovering raw materials from waste products is obvious.

3. Experimental

3.1. Facility and materials

The plant for heavy metal oxide and oxide mixture recovery mainly consists of the following components:

- evaporation plant for the liquid input materials,
- rotary kiln,
- hot gas filter,
- scrubber column,
- dioxin filter.

The structure of the pilot plant at the Aue site is shown in Fig. 3.

The chief component of the plant is the hot gas filter, which comprises vertically arranged ceramic filter candles.

During the preliminary experiments, the functionality of the plant was tested by the throughput of a solution containing sodium chromate tetrahydrate ($\text{Na}_2\text{CrO}_7 \cdot 4\text{H}_2\text{O} + \text{H}_2\text{O}$). To produce the test solution, approximately 16.25 kg $\text{Na}_2\text{CrO}_7 \cdot 4\text{H}_2\text{O}$ was dissolved in distilled water. The total volume of the solution was 35 l, so that the chromium content was about 8 wt%.

3.2. Process

In order to increase the process's economic viability and to make it more energy-efficient, an evaporation device was

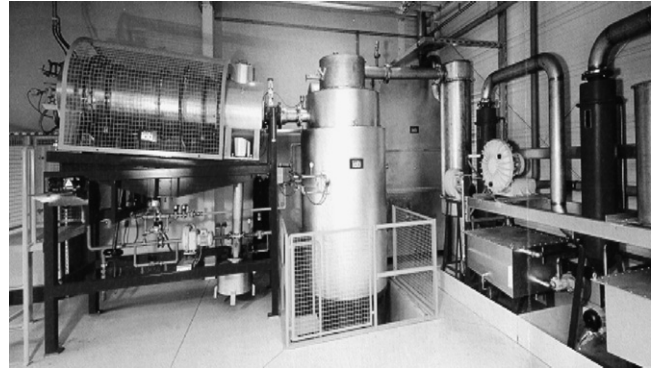


Fig. 3. Structure of the plant.

integrated upstream of the actual plant. This enables both the liquid input materials to be concentrated (leading to throughput enhancement) and the residual heat emitted to be optimally utilized. Once the materials had been injected via nozzles into the rotary kiln by means of a natural-gas burner, the concentrated substances were heated up to a temperature of 900 °C. The experiments showed that the components containing ammonium chromate in the solution undergo reduction to chromium(III) oxide (Cr_2O_3) in the sub-stoichiometric environment. The oxygen released during the reaction is used as an oxidant for the natural gas, ensuring that reducing relationships always prevail in the reaction zone. The following figure summarizes the reactions taking place.

In addition to the corresponding reactions, the main relationships between the individual processes are illustrated in Fig. 4. The connection between the oxygen resulting from the chemical process and oxygen consumption during the combustion process is especially apparent.

A Lambda probe made by Metrotec GmbH and installed between the rotary kiln and a hot gas filter measures the level of oxygen in the plant. Measuring the Lambda value in sub-stoichiometric ranges forms the basis for the plant control system, which was planned by PMA.

Regarding the operational parameters of the plant, the following control variables need to be determined for the process:

- amount of input solution,
- supply of primary air (addition of combustion air),
- pressure below atmosphere in the system.

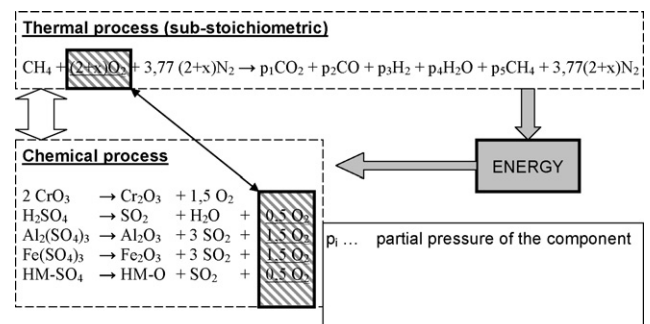


Fig. 4. Processes.

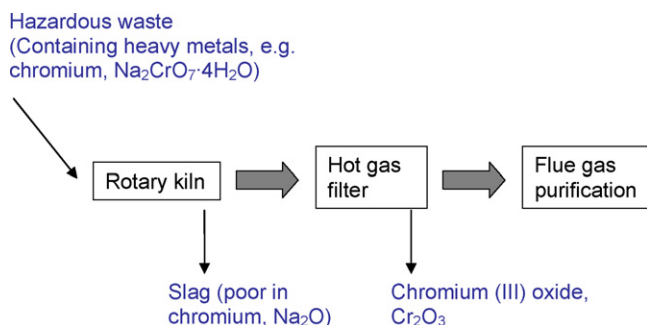


Fig. 5. Flow chart of the processes.

Due to the very small grain size of the Cr_2O_3 formed, the chromium remains in the flue gas while other compounds and elements such as sodium remain in the slag. This fulfils the first aim, i.e. separating chromium and sodium.

The isolation process of the product Cr_2O_3 (the actual separation of the product from the flue gas current) takes place in the hot gas filter at the core of the plant. Cr_2O_3 can, for example, be used as an anti-corrosive agent in the aircraft industry.

After having passed the acid or base scrubbing column and the dioxin filter, the purified flue gases escape the plant via a stack. This process is illustrated in Fig. 5.

3.3. Sampling and analysis of the reaction products

Analysis focused on the input flows (for the purpose of plant protection) as well as the slag and filter dust. The idea was to demonstrate the principal reaction mechanism and to determine the balances of materials' flows.

The entire analysis of the liquid input materials and also the solid reactants relied on X-ray fluorescence analysis. The essential advantage of XRF was the simple preparation of the samples. In this regard, the liquid samples (input materials) could be directly sampled, and the solid fractions (filter dust and slag) were measured both in powdered form and in terms of tablets. Consequently, digestion of the samples was unnecessary.

However, following comprehensive calibration (development of methods) of SPECTRO's XEPOS XRFA unit, it was found that the analytical method developed was only partly suitable for the exact quantification of elements (especially heavy metals) in trace amounts. Because this analytical method is indispensable, primarily for the quality supervision of the filter dusts formed, the filter dust was additionally examined after microwave digestion using atomic absorption spectrometry. Furthermore, the respective input flows were examined by means of ion chromatography for their content of chromate. During the experiments the filter dust was analyzed as follows:

- chromium, silicon (total) → X-ray fluorescence analysis,
- sodium, magnesium, aluminum, iron (total) → microwave digestion + AAS.

Table 3
Measurements of filter dust composition

Silicone (total) (wt%)	1.77
Sodium (total) (mg/kg DM)	422
Magnesium (total) (mg/kg DM)	27
Aluminum (total) (mg/kg DM)	398
Iron (total) (mg/kg DM)	138
Chromium (total) (wt%)	61.15

DM: dry matter.

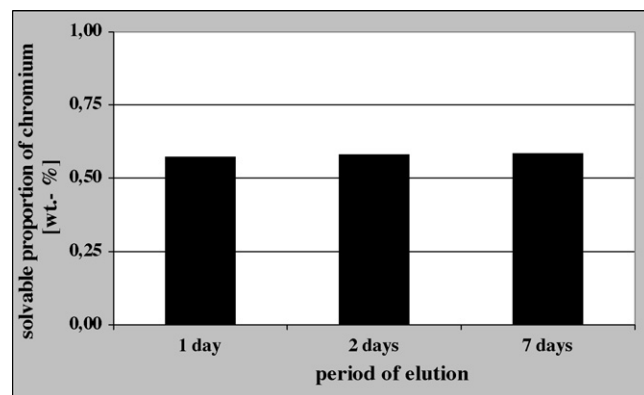


Fig. 6. Results of the solubility experiments.

3.4. Results

The results of the investigations are summarized in Table 3. On the one hand the accumulation of chromium and on the other hand the almost complete separation of the input elements sodium (422 mg/kg DM) and chromium (61.15 wt%) are obvious. The significant proportion of silicone is attributable to the phenomenon of erosion in the rotary kiln. The trace elements iron and aluminum similarly result erosion in the area of the pipes.

To investigate the type of chromium bond as well as the conversion of water-soluble sodium chromate into chromium(III) oxide, approximately 1 g of the sample each was mixed with 11 of distilled water in subsequent experiments and the solutions were examined by ion chromatography for their level of chromium. Fig. 6 illustrates that even after 7 days' dwell time of the sample in distilled water, there was hardly any chromium (0.34 wt%) in the aqueous phase. It can thus be assumed that the sodium chromate tetrahydrate had been almost completely converted into chromium(III) oxide with the aid of the plant. Hence, assuming that the element chromium is available in the filter dust as chromium(III) oxide, based on stoichiometric computations a level of chromium(III) oxide exceeding 90 wt% in the filter dust can be determined.

4. Conclusions

During the project for the thermal treatment of hazardous waste for heavy metal recovery, the optimization process involved the construction of a pilot plant in which the method was proven to work. The verification of chromate reduction (subsequent to the analysis of filter dust using ion chromatography

Cr(VI) content <0.5 wt%) to Cr₂O₃ demonstrated the occurrence of an essential step for optimizing the procedure for the recovery of heavy metal oxides and oxide mixtures. In order to avoid the input of silicone contaminants, the rotary pipe was replaced after the preliminary experiments.

If the procedure described can be optimized, it will provide a way for the safe, reliable and above all high-grade utilization of hazardous waste materials. Because of the good sales opportunities (for instance, the filter dust formed being purchased by the aircraft industry), the economic aspect may supposedly be integrated as well. According to calculations operating costs amount to 15–20 percentage of current market price.

Safe plant operation and quality control entail the systematic sampling of the respective mass flows. The ideal solution in this respect appears to be to use X-ray fluorescence analysis combined with atomic absorption spectrometry.

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