Froth Flotation, as a Remediation Process, Applicable to the Treatment of High Mercury Solid Waste

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Abstract-The aim of this study was to create a basis for a reliable, efficient and economic process for the treatment of high mercury solid mineral waste, applicable particularly for soil from former chlorine alkali electrolysis plants and acetaldehyde factories but also for various sorts of mercury-containing sludge such as commonly generated from the oil and gas industry. Alongside the removal of mercury from the contaminated soil by using Sodium Dimethyldithiocarbamate (SDMC) as an oxidation agent and Potassium Amylxanthate as a flotation collector and complexation agent. The process also uses the advantage that $Hg2+$ reacts with xanthates to create very stable and water insoluble square-planar complexes. Consequently, both the "cleaned" mercury-lean fraction (tailings) and even the mercury-enriched effluent represent finally, a leach-stable material capable for safe and environmentally friendly disposal.

Keywords: High mercury waste, flotation process, chlorine alkali electrolysis, Hg(II)-thiolate complexes, soil washing, remediation technology

I. INTRODUCTION

At present large scale ex-situ remediation processes for the effective and economic treatment of mercury-contaminated solid waste - such as soil from chlorine-alkali electrolysis plants but also mercury-contaminated sludge such as generated from the oil and gas industry - are limited to stabilization/solidification (S/S) [1] [2][3][4][5] and thermal desorption, which as per US Land Disposal Restrictions Regulations (LDR) is understood as "retorting – RMERC". Retorting is recommended particularly as a remediation method for "high mercury inorganic waste" with Hg > 260 mg/kg [6]. However, the application of S/S to mercury-containing waste is limited, owing to negative effects due to necessary sulfidization agents and associated contaminants such as hydrocarbons, which are not compatible to the commonly applied hydraulic binders. Also, S/S treatment is only recommended for low mercury waste (Hg < 260 mg/kg) according to the US Land Disposal Restrictions[6] consequently making thermal desorption the most commonly used and widely accepted technology for the treatment of highly contaminated mercury waste. The problem concerning high mercury waste such as mercury-contaminated soil, is an issue affecting essentially all industrial countries world-wide which previously applied particularly chlorine alkali electrolysis in the mercury cell (amalgam) process (aka Castner-Kellner Process), had acetaldehyde factories or were operating chemical/pharmazeutical factories producing Hg-containing pigments, medication or seed preservants.

Thermal desorption as a treatment technology for mercury-contaminated soils has not improved significantly in the last ten years, especially after the remediation of the Marktredwitz Chemical Factory (CFM)/Germany in June 1996. The Marktredwitz Plant, particularly the thermal treatment unit, was constructed and commissioned under the supervision of the author in 1992 and 1993, as a unique 'prototype plant' at this time. It was used to successfully treat about 56,000 metric tons of mercury-contaminated soil and demolition debris from an inner city abandoned chemical factory in the Franconian Town of Marktredwitz.

The Chemical Factory Marktredwitz was founded in the year 1788. In spite of the fact that the area formerly belonged to the Austrian Empire, it is considered to be the oldest chemical factory in today's Germany. The treatment plant, which was located outside of the town of Marktredwitz, comprised "soil washing" combined with an indirect fired thermal desorption (called: vacuum distillation) process unit (shown in Fig. 1). Inopportunely, in the English literature the Marktredwitz Plant is often misunderstood as a pilot-scale plant only [7] and therefore not very well-known.

More so indirect fired thermal desorption facilities for mercury waste treatment have major mishaps such as limited material throughput, handling of excessive dust generated from the thermal destruction of the soil particles (particularly silt and clay), corrosion issues and the re-condensation of the mercury from the dust-loaded off-gas stream creating mercury bearing waste that also requires further treatment [8]. The most pressing short coming however is the higher treatment price at a comparatively low throughput rate and the lesser technical availability rate compared to equal alternative technologies such as enhanced soil washing [9]. However, thermal desorption (vacuum distillation) remains the preferable treatment technology for high mercury waste with Hg greater than 1.000 mg/kg DM (dry mass).As it is not possible, in a conventional soil washing process, to transfer the mercury contamination (particularly metallic mercury) from the sand fraction into the fines (clay and silt) and enrich it in the filter cake such as it is commonly practiced while dealing with other contaminants [10], it is necessary to up-grade the currently available facilities with efficient pollutant sinks such as flotation [11] in order to treat mercury wastes even up to ranges of 1,000 mg/kg DM with wet-mechanical processes such as soil washing.
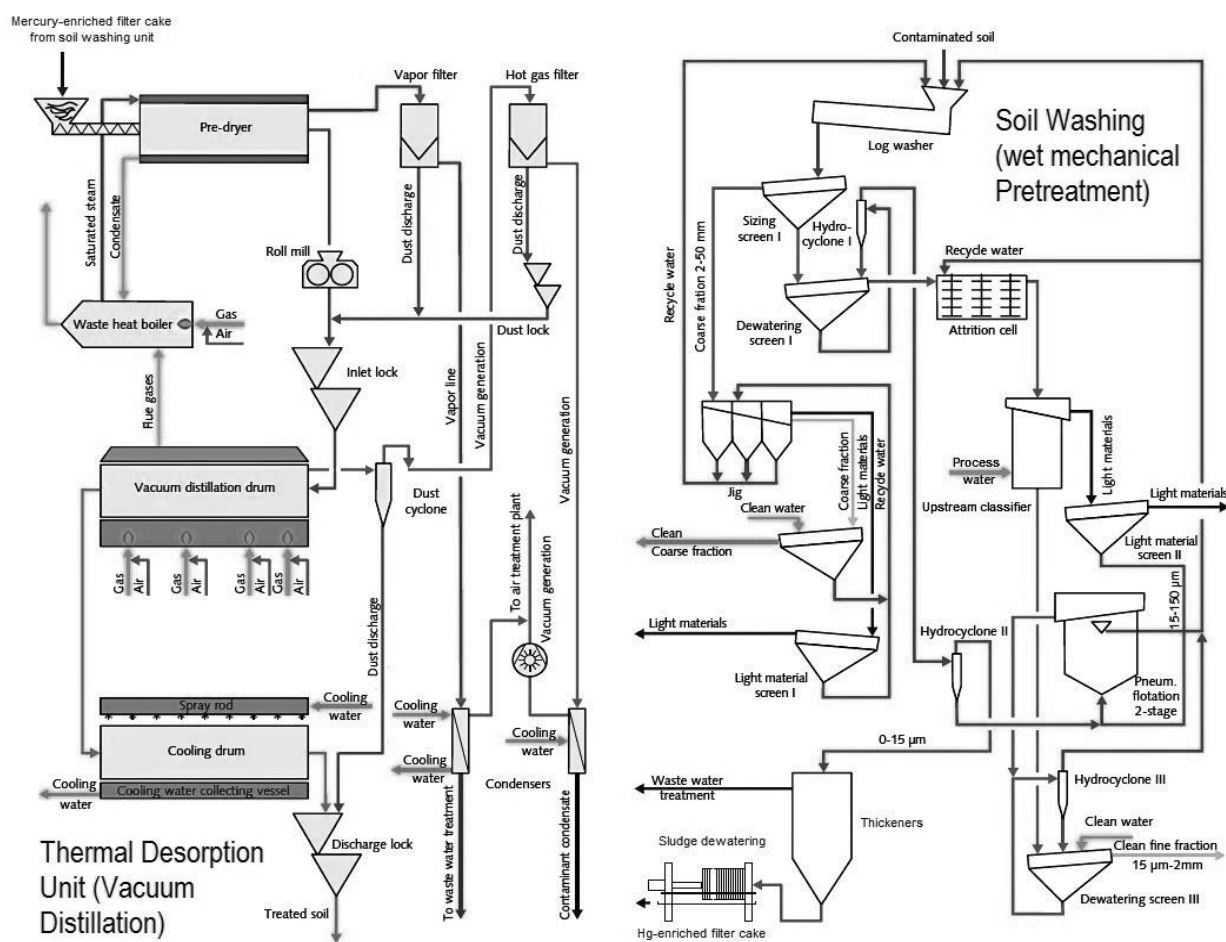

Fig. 1: Process Diagram of the "Marktredwitz Plant" for Hg-contaminated soil and demolition debris from an abandoned chemical factory (Chemische Fabrik Marktredwitz).

Flotation as a separation process for mercury-containing soil was already subject of US patent 5,244,492 A [12], where flotation was used to separateinsoluble (visible/metallic) mercury only. In the subsequent described novel treatment process by flotation, the insoluble mercury (commonly available Hg0) in a controlled pH range between 8 and 10.5 is turned in a first step into a soluble mercury compound in the form of the sulphur mercuric ion (HgS22-) by a reaction with Sodium Dimethyldithiocarbamite (SDMC). Then all the highly soluble mercury compounds are immobilized by Potassium Amyl Xanthate (PAX) by forming insoluble mercurous xanthates (a leach stable square planar complex). PAX, aka KAX as per Taggart's nomenclature [13] also acts as the collecting agent. This entire process is further on referred to as "the new flotation process" in this paper.

The more recent published applications of flotation techniques or adsorption processes pertaining to the treatment of mercury-contaminated wastes are mostly constrained to 'Ion Flotation' by dissolved air flotation apparatus, aka DAFs [14] and some very specific technical applications such as the removal of Thiomerosal from industrial process water [15] or the removal of mercury cyanide complexes from the processing streams of gold hydrometallurgical operations [16]. Next to required coagulants and auxiliary flocculants, Dithiocarbamates (R–N (–R″)–C (=S)–S–R′) are used in these applications for precipitation, only. Paraffin and 0.1 M Potassium Ethyl Xanthate (KEtX) were used as collectors for metals such as Zn, Pb, and Cd from sediments of the Gent-Terneuzen Canal by Vanthuyne and Maes [17].The highest metal recoveries could be achieved at pH 12 showing slightly better results with kerosene as collecting agent (between 80 and 90 % for particularly Cd) compared to KEtX. Earlier performed flotation tests with Gent-Terneuzen Canal sediments have shown that the results of flotation tests performed with the whole sediment range (< 2 mm) were much better than those for the fine particle fraction only. This effect also was reported from earlier performed industrial scale flotation tests with metal-contaminated soils in the physico-chemical solid waste treatment plant Berlin-Gradestrasse by Stapelfeldt and Richter [18].

This paper illustrates experimentally the applicability and effectiveness of the 'new flotation method' for the treatment of high mercury contaminated wastes such as soils from chlorine-alkali electrolysis plants and for mercury contaminated sludge. Previous experiments on flotation of heavy-metal contaminated soils from a previous inner city steel works and rolling mills site in Berlin showed recoveries of maximum 75 % for lead and 44 % for zinc obtained for a one step flotation in laboratory scale. The recovered cleaned soil amounted to 92 M-% [18]. Results showed that a recovery of mercury from fines (clay and silt; 25-250 μm) under the given laboratory conditions is possible and therefore prompted trial tests with an artificially mercury-contaminated soil (Hg = 1,000 mg/kg and without any prior activation/oxidization) were conducted. The best decontamination results were achieved with Potassium Amyl Xanthate (PAX aka KAX) in a range of 400 ± 100 mg/kg and at a pH range between 8 and 10.5.

1.1 Soil Preparation, Characterization and Experimental Methodology

1.1.1 Preparation Soil for Flotation Tests

In order to achieve comparable results, soils artificially contaminated with variable amounts - 800 mg/kg, 1,000 mg/kg and 1,200 mg/kg (all concentrations are related to dry mass) - of beaded (metallic) mercury were used. All samples also contained chloride in a range of 200 mg/kg, simulating a typical mercury-contaminated soil from a chlorine-alkali electrolysis plant.

In addition, soil from an ongoing mercury remediation project, which previously had been sampled by the author from the 'Spolchemie' Chemical Factory (Spolek pro chemickoua hutní výrobu) located in Ústí nad Labem (Czech Republic), was prepared for anthropogenic (original) contamination consideration.

Furthermore, additional mercury was added to some samples in order to reach contamination levels comparable to the purely artificially generated test soil hence making it three sample sets. (see

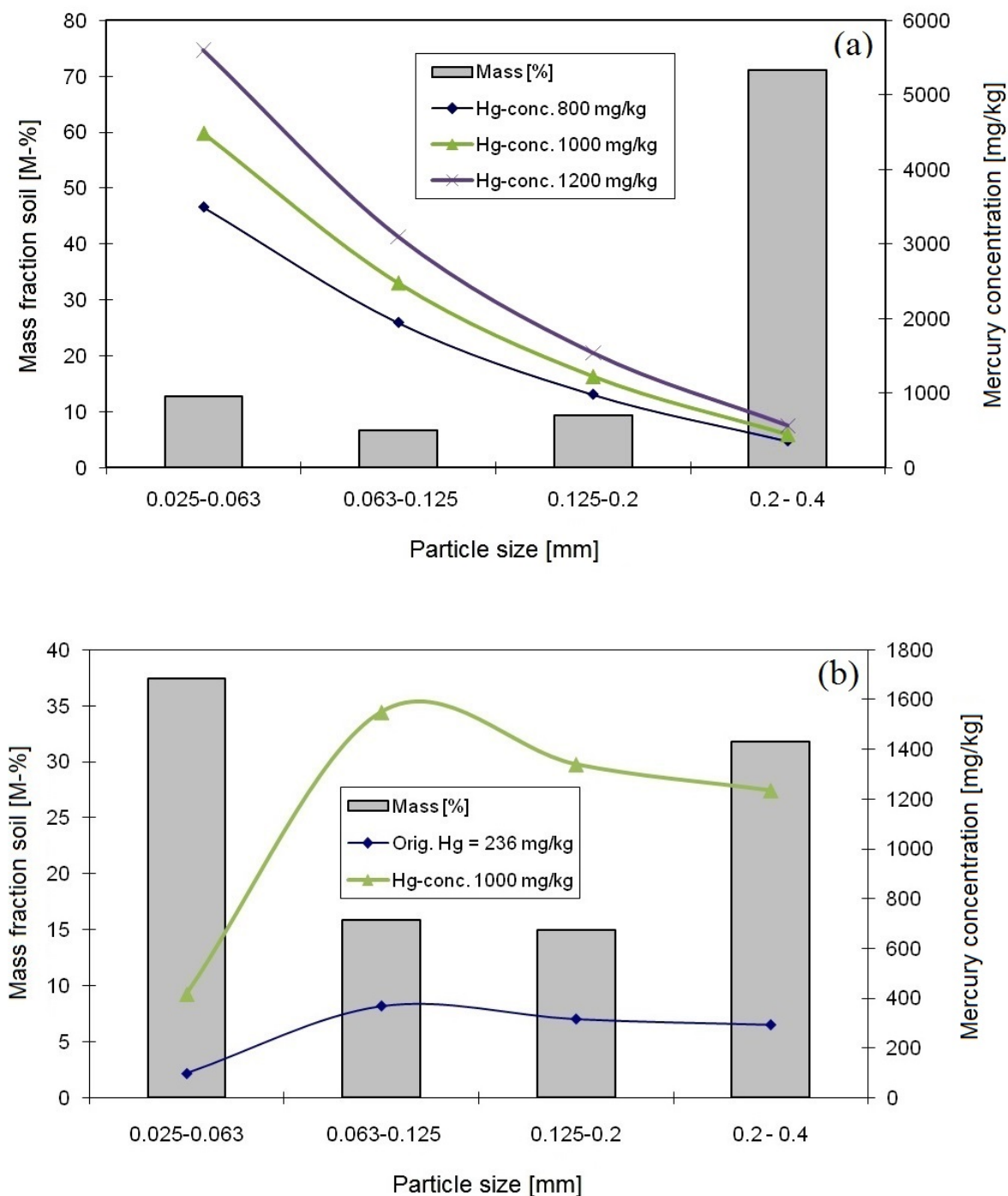Fig. *2* below and

TABLE **1**).

Fig. 2: Concentration of Hg in different particle size groups of test soils with 3 different degrees of Hg-contamination; (a): from artificial contamination and (b): from the Ústí nad Labem chlorine plant.

TABLE 1: Soil From ÚstíNadLabem Prepared For Flotation Tests

Particle size [mm]	Weight pass [g]	Weight pass [%]	Orig. Hg-conc. [mg/kg]	Art. Hg-conc. [mg/kg]
0.025 – 0.063	52.20	37.34	98	415
0.063 – 0.125	22.20	15.88	368	1,550
0.125 – 0.200	21.00	15.02	316	1,340
0.200 – 0.400	44.40	31.76	293	1,235
total	139.80	100.00	236	1,000

1.1.2 Characterization of Typical Soil from Chlorine Plants

The mercury-contamination in soil, but also in the demolition debris of former chlorine alkali electrolysis plants (CAPs) typically varies in a wider range. As an example results from soil investigations have been performed with soil samples from various CAP areas the Buna Works in Schkopau [18].

TABLE 2:Mercury Contamination Analyzed In CAP Soil From The Buna Works Schkopau / Germany (GKSS)

Chlorine Plant Area	Total mercury [mg/kg] DM	MeHg+ [µg/kg] DM	MeOEtHg+ [µg/kg] DM
Buna H56	190 – 3.000	4 - 48	Not determined
Buna I 54	45 – 550	8 - 14	1 – 17
Buna L66	100 – 5.900	24 - 400	3.5 – 18

In soils contaminated with mercury biological processes result in organic mercury compounds with alkyl or acryl groups such as the methyl mercury anion $[CH_3 Hg]+$ and diphenyl mercury $Hg(C_6H_5)_2$. The ratio of methyl mercury compared to the total concentration of Hg in sediments and soils is commonly between 0.1 and 1.5 %, which is affected by the balance of methylation and de-methylation [19].

Contaminated earth from the remediation of former chlorine alkali electrolysis facilities, particularly in case of sandy soils, contains next to mercury also considerably high concentrations of weathered carbon (visible grains) such as from the graphite electrodes (maintenance residues) but also from other sources such as metamorphosed wood. Iron in such soil can occur from various corrosion products such as in particular as iron hydroxides (Goetheite), oxides or carbonate is also a possible adsorbent for mercury. These mercury-contaminated fractions can be removed by density separation processes which are recommended as additional pollution sinks to be established in soil washing plants in parallel to flotation as it was demonstrated in a soil washing center in Berlin (Germany) by Richter, Schmidt and Stapelfeldt in 1997 [20] and just recently by Hirsch, Rammer, Richter, Flachberger and Kabogozza [21]. The chlorine plant soil from Ústí nad Labem (Fig. 2b) did not contain visible particles of coal or corroded iron, related to the mainly loamy character of that sample but the same soil showed in some locations excessively high concentrations of sodium from process losses. Alkali metals, particularly sodium or potassium, tend to form amalgams with mercury which are unstable in an aqueous environment, which might be different in saline-sodic clays such as in soil from Ústí nad Labem.

1.1.3 Mercury Sulfide Species in The System Soil-Water

In wide pH- and redox potential ranges the elemental form of mercury Hg0 prevails. Depending on the redox potential and complexion agents the elemental mercury is oxidized to Hg2+ by exposure to air or oxygen-containing water. Particularly chloro complexes and sulfides are to be mentioned here.

The occurrence of mercury sulfide and other inorganic mercury species depends very much on the pH and the solubility, but also whether the aquatic system is operated under oxic or anoxic conditions. This is significant for the understanding not only for the sufficient application of flotation technologies on high mercury wastes and for the S/S process applied to such wastes, but also for the methylization process taking place in mercury-contaminated soils in general.
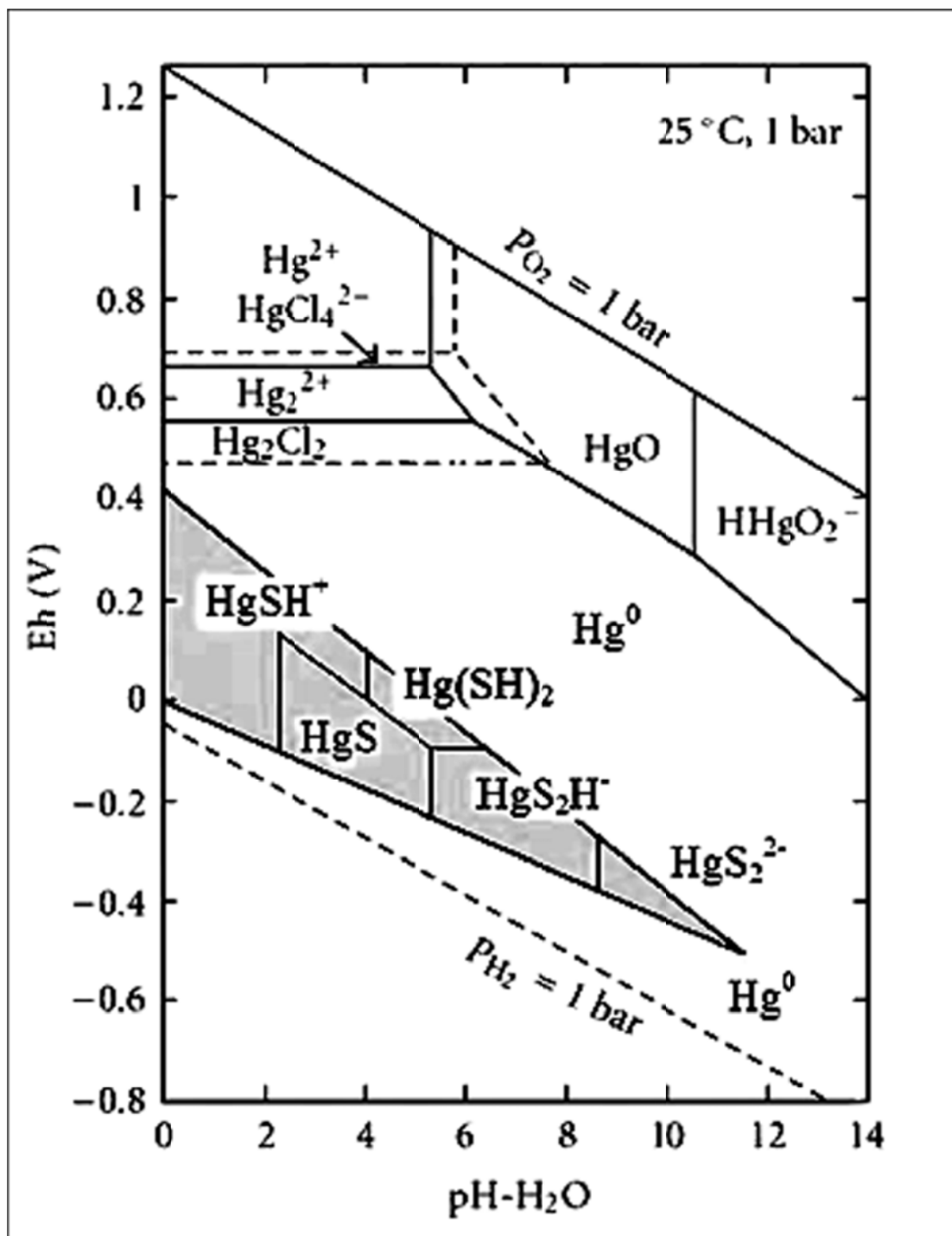
Fig. 3: Eh-pH diagram (simplified) shows dissolved mercury species in the system "Hg:S: Cl = 0.5:1: 1"

Fig. 3 shows various dissolved mercury species in the system: "Hg: S: Cl = 0.5:1: 1". This system application is very good for soils from former chlorine-alkali electrolysis plants, owing to the existence of chlorine ions from process residues introduced to the ground by incidents or leakage. On the other hand, soil (rubble anyway) of former chlorine plants is in the majority of all remediation cases in the low alkaline range, mostly caused by Sodium ($Na+$) or Potassium ions ($K+$), but also by their hydroxides. Owing to this the preferable pH range for processing mercury-contaminated waste, solid materials shall be above 8, accordingly.

From the three known solid HgS(s) species such as red cinnabar, hexagonal hyper-cinnabar and black meta-cinnabar, in an aqueous system predominantly amorphous meta-cinnabar may be formed by the reaction of Hydrogen Sulfide with dissolved mercury compounds. The very important mercury sulfide compounds disappear completely in the system: "Hg: S : Cl = 5 : 1 : 1" according to Svensson [22], considering metallic mercury as the dominating species, which is also one of reasons for the technical limitation of wet-mechanical treatment technologies.

1.2 Equipment Used and Execution of the Flotation Program

The main flotation tests were performed within a standard Denver D 12 laboratory flotation machine at a constant rotation speed of 1,600 1/min and a 2-liter glass cell. The only varying parameter was the concentration of the activation/oxidization (sulfidization) agent, Na-Dimethyldithiocarbamate (SDMC), which was added in steps, starting with a concentration of 0.5 g/kg up to finally 8 g/kg. The oxidation effect of thiol compounds on dissolved elemental mercury under anoxic conditions was confirmed also by Zheng et al., 2013 [23].

TABLE **3**: FLOTATION PROGRAM (COURSE SCHEME OF THE FLOTATION TESTS)

Step/activity performed	Description/used reagent	Duration [s]
Dispersing	400 g of soil of a grain size range 25-500 μm is dispersed in a 2 l flotation cell (pulp density 200 g/l)	120
Oxidation of Hg^0 to Hg^{2+}/Sulfidization	SDMC is added in concentrations between 0.5 g/kg and 8 g/kg.	900
Addition of collector	KAX = 400 mg/kg (const.)	120
pH adjustment	adjusted in a range of pH 8 up to 8.5	-
Addition of frother	Flotanol D 12 was added drop wise (appr. 0.1 g/kg)	20 (approx.)
Flotation	Flotation started, when froth developped	300
Dewatering effluent	Effluent containing Hg sulphide was dewatered in a suction strainer supported by a water-jet vacuum pump using a standard laboratory round filter	-
Drying of tailings after dewatering	Pre-drying occurred under the fume cabinet slowly over night. Afterwards for 1 h in the drying oven at 42 ºC.	12 h
Weighing and packing of samples	Weighing was performed on a Mettler laboratory high precision scale and samples packed for analysis afterwards	-

More important than the chemical characteristics and the correct dosage of the oxidation agent is the adjustment of the pH, which shall be exactly in the range, where mercury sulfide species are soluble (8.5-11.8) The course scheme of the flotation tests is given in
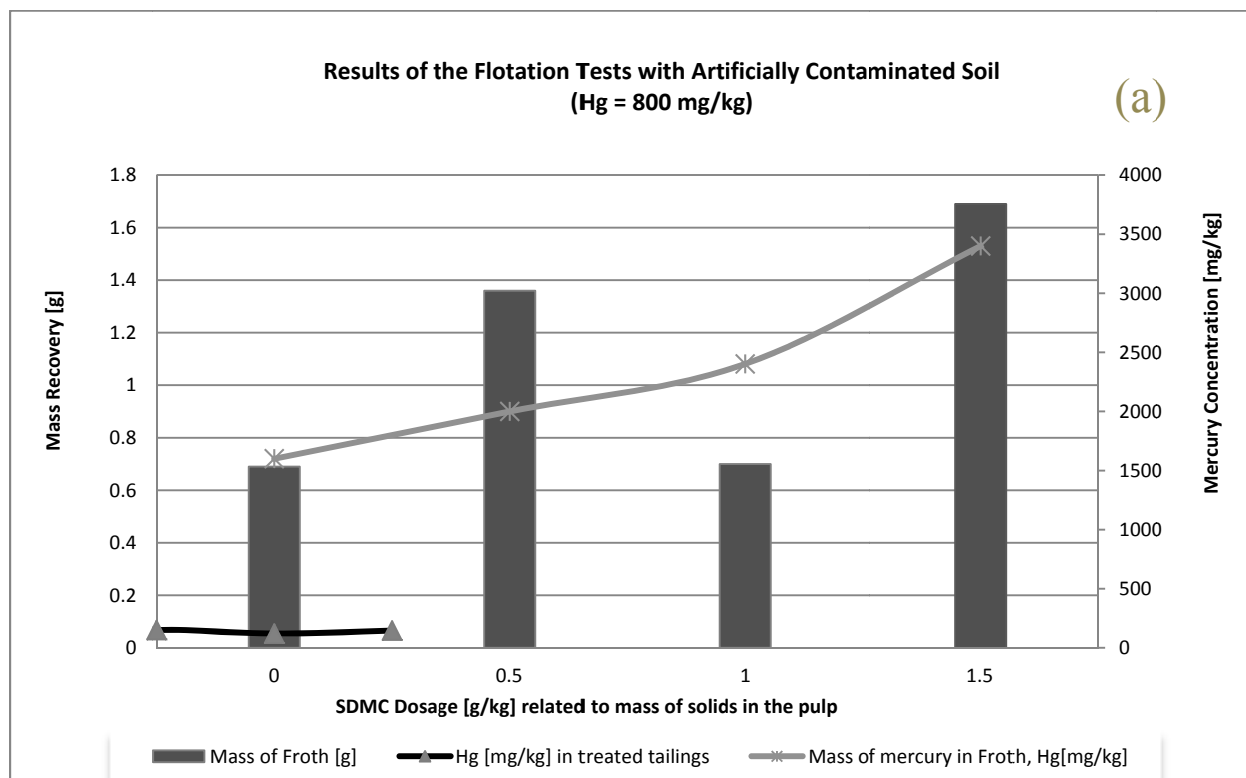
TABLE .

II.   RESULTS AND DISCUSSION

In the following section the results of the flotation tests with the above described artificially Hg-contaminated soil and the material from the Ústí nad Labem chlorine plant.are presented and discussed.

2.1 Results of Flotation Tests Performed with Artificially Hg-Contaminated Soil

The best test result with regard to mercury in the original substance could be achieved with a SDMC dosage of 2 g/kg, based on the solid content in the pulp. Here the mercury concentration in the mercury-contaminated soil sample could be reduced from Hg = 1000 mg/kg to Hg = 4 mg/kg, at most. Other results of the same test row show values merely above the threshold value Z2 (10 mg/kg) as per German LAGA regulations.

A graphical representation of some of the data is shown in Fig. (a), and (c) below;

Results of the Flotation Tests with Artificially Contaminated Soil (Hg = 800 mg/kg) (a)
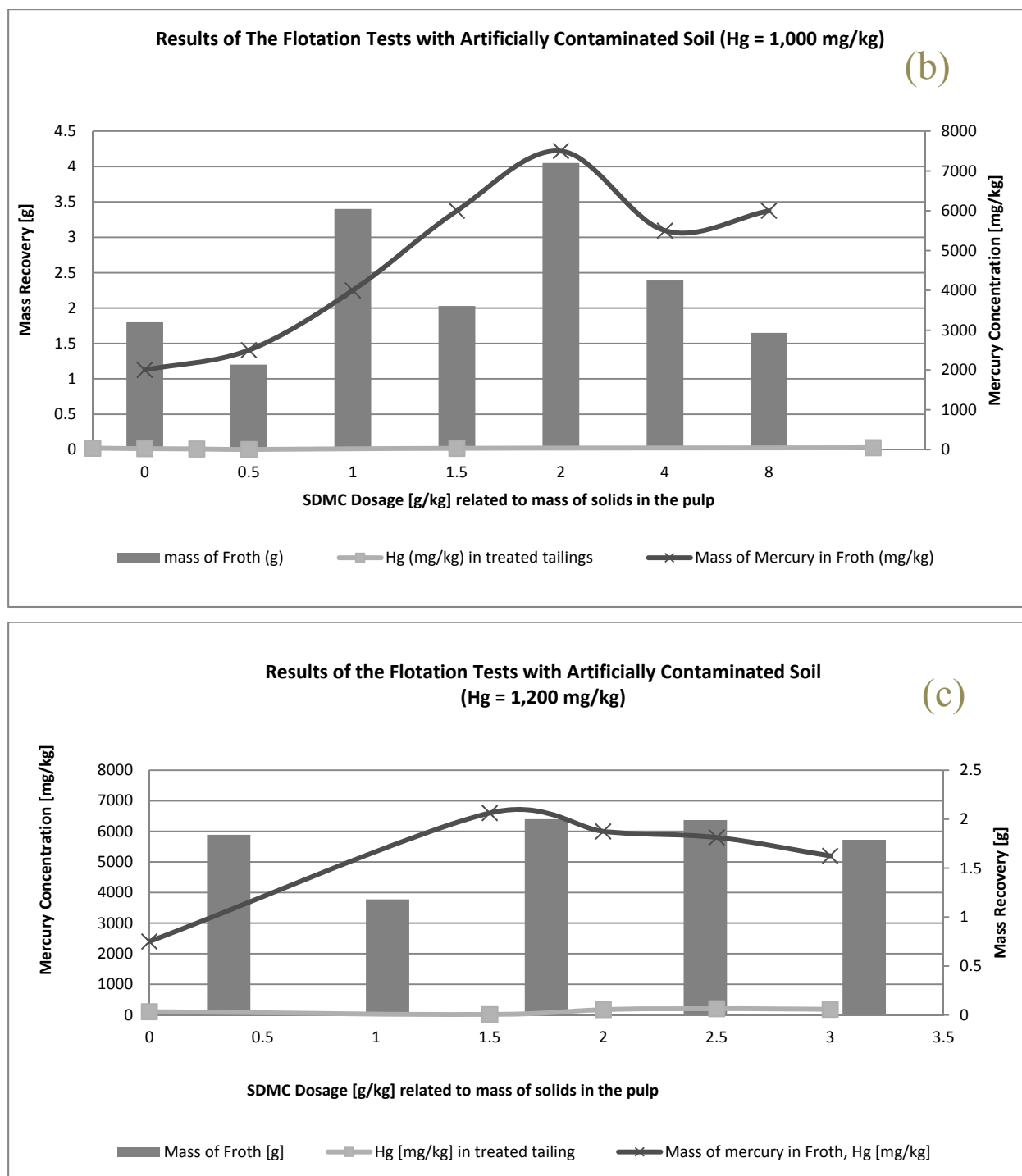
Fig. 4: Results of flotation tests with artificially contaminated soil (a); Hg = 800 mg/kg, (b); Hg = 1000 mg/kg, (c); Hg = 1,200 mg/kg.

The laboratory flotation test was performed only in one single cell, however, it is most likely that if sub aeration cell flotation is implemented in a soil washing plant, as it is the common practice with 6 or more cells (cell-to-cell transport in series) would lead consequently to even much better results.

2.2 Interpretation of TCLP Test Results with Artificially Hg-Contaminated Soil

To verify the applicability of the 'new flotation process' performed in the course of this study the tailings were tested by Toxicity Characteristic Leaching Procedure (TCLP) as per EPA Test Method 1311 (Fig.5, below) [24].
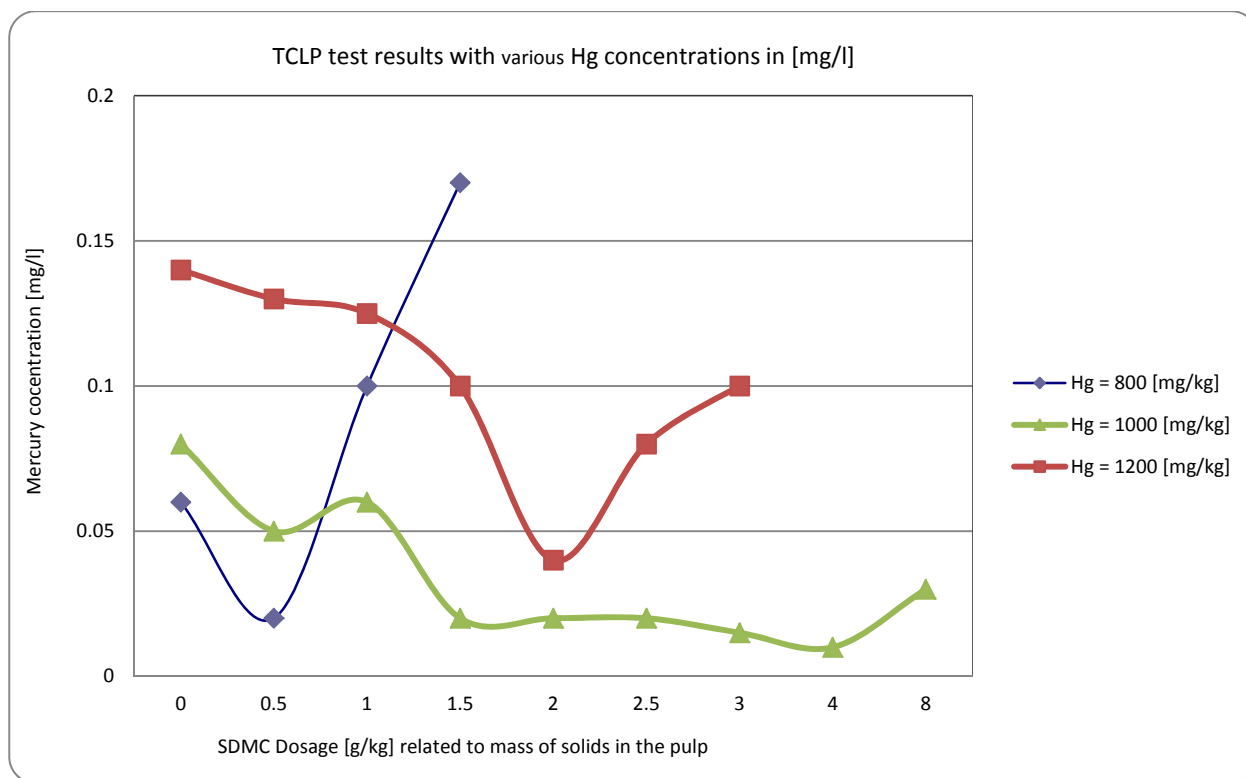
Fig. 5: TCLP test results of treated soil (tailings) performed for (a); Hg = 1,000 mg/kg, (b); Hg = 800 mg/kg, (c); Hg = 1,200 mg/kg.

The TCLP-test results in Fig. 5 demonstrate that the flotation tests performed are entirely successful because none of the results exceeded the associated D-list (hazardous) parameter for mercury (Hg = 0.2 mg/l) in any single result (US EPA Method 1311, 1992). A more complete complexation of mercury with the collecting agent KAX, such as shown in Fig. 6, could be achieved at a mercury concentration of 1,000 mg/kg compared to 1,200 mg/kg, which also is related to the ratio of Hg:S: Cl (Fig. 3).

The optimum SDMC dosage, where such leach-stable complex is formed at a mercury concentration of 800 mg/kg is 0.5 g/kg, whilst the best result for the 1,200 mg/kg line is achieved with SDMC = 2 g/kg. This effect occurs, because surplus SDMC (more than it is required for oxidation) also complexes with mercury and obstructs the formation of a leach-stable square planar Hg(II)-Amyl-Xanthate Complex (Fig. 6).

2.3 Results of Flotation Tests Performed with Soil from ÚstíNadLabem

The second test soil from Ústí nad Labem, with a content of fines (silt and clay) in a range of almost 40 M-%, is not applicable for a conventional soil washing process (without flotation) [10][11]. That is the reason why currently a thermal treatment is applied to the site. With the original contaminated soil, a decontamination effect from the original Hg = 236 mg/kg to 26 mg/kg could be achieved, which is just below the site treatment limit of 30 mg/kg, as requested by the environmental authorities for that site remediation.

The flotation test with the artificially increased mercury concentration soil from Ústí nad Labem did not fully succeed, since the decontamination effect from 1000 mg/kg to 126 mg/kg was low. With a multistep flotation system (cell-to-cell transport) the results could be improved, accordingly. However, all leachability tests performed with treated soil in laboratory scale passed the leaching criteria for Hg < 0.2 mg/l as regulated per US EPA (US EPA Method 1311, 1992) [24] which would allow a disposal in a hazardous waste landfill.

One reason for the less efficient treatment of the real chlorine plant soil compared to the artificially contaminated sand is most probably the higher salinity and the higher concentration of sodium in the clay (sodic clay). Salinity (chlorine concentration) affects the appearance of the sulfur mercuric ion HgS22- as the most important soluble sulfurate mercury species. Sodic clay could have an impact owing to the formation of more long living amalgam complexes.

2.4 Discussion of Results Achieved and Interpretation of the Novel Flotation Process

The occurrence of a variety of mercury species depends very much on the pH and the solubility, but also whether the aquatic system is operated under oxic or anoxic conditions. This is significant in understanding not only for the sufficient application of flotation technologies on high mercury wastes and for the S/S treatment process applied to such wastes, but also for the methylization process taking place in mercury-contaminated soils in general.

TABLE **1** shows the solubility (at 20 oC) of these sulfur-mercury complexes. The sulfur mercuric ion HgS22- as the only soluble sulfurate mercury species is of great significance for the success of currently applied mercury technologies.

TABLE **4:** SOLUBILITY PRODUCTS OF SOLID MERCURY SULFIDE SPECIES [25][26]

Mercury-sulfide species/ reaction	logS	K_{sp} Solubility product $[(mol/l)^3]$	$logK_{sp}$	Equilibrium constant log K_f [mol/l]	pH range
$HgS_{(S)} + H_2S \Leftrightarrow HgS_2H_2$	-5.97	4.928×10^{-18}	-17.31	37.5	4.0 – 6.2
$HgS_{(S)} + HS^-_{(aq)} \Leftrightarrow HgS_2H^-$	-5.28	5.78×10^{-16}	-15.24	32.0	5.5 – 8.5
$HgS_{(S)} + S^{2-} \Leftrightarrow HgS_2^{2-}$	+0.57	205.15	+2.31	23.5	8.5 – 11.8

The xanthates of class b and borderline metals such as mercury, silver, copper and lead are more or less insoluble in water. In particular, xanthates form very strong complexes with certain class b metals such as mercury. The xanthates of zinc, iron and manganese are moderately soluble. Aurous xanthates, in particular gold alkyl xanthates, were synthesized by Denko and Anderson, 1945 and described as insoluble in water [27]. Different solubility properties of heavy-metal xanthates reflect, to a certain degree, chemisorption on the mineral surface, and explain the effect of selective collection of xanthates. In addition, the length of the alkyl group has a considerable influence on the solubility [28].

Concerning mercuric xanthate, Koten and Adams,1924 published their research results on alkyl and aryl mercuric hydroxides and their article is one of the most remarkable early articles dealing with alkyl mercuric xanthates R'HgS(S) C.OR and aspirational until today [29]. The reaction had been catalyzed by sodium hydroxides in order to prevent the formation of mercaptans.

Casas et al., 2002 [30] recently performed a research on the crystal structure of methyl mercury MeHgS (S) C.OR. The more interesting interpretation of the results is that xanthates are able to immobilize highly soluble and extremely toxic methyl mercury compounds into insoluble mercurous xanthates, which qualifies xanthates as the preferable collector for the treatment of solid wastes from chlorine alkali electrolysis and acetaldehyde factories.

Owing to the varying solubility of various mercuric sulphide species in aqueous systems a flotation method was developed which finally formed highly stable square-planar insoluble mercury complexes with the collecting agent even in the flotation foam product (effluent).

The main advantage of the process is the leach-stable fixation of the mercury contaminant in square-planar complexes of such as shown in the ORTEP-diagram (**Error! Reference source not found.**6). In order to apply this configuration, the commonly available metallic mercury Hg0 needs to be oxidized to Hg2+ prior to the final reaction with the collecting agent:
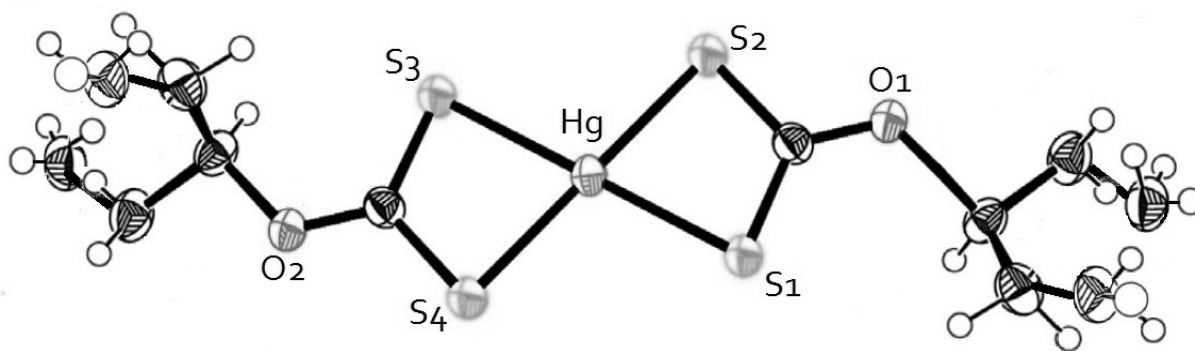

Fig.

6: Possible ORTEP structure of the generated Hg(II)-Amyl-Xanthate Complex

2.5Re-Use and Disposal of the Output Materials and Comparison with Other Treatment Methods

The cleaned sand (flotation tailings), after undergoing the described froth flotation process, can be re-used at least as capping material for landfill sites or, depending on the remaining mercury content and on the country's individual legislation, even as a construction material. Since the mercury in the enriched froth product is bound into a leach-stable complex it can be disposed easily in a double lined hazardous waste landfill (Hg < 0.2 mg/l) such as recommended by the US EPA. Compared to thermal processing, particularly in terms of treatment costs, soil washing enhanced by flotation, as a treatment method for highly mercury-contaminated solid waste (up to max. 1,000 mg/kg) has certain advantages. The processing in the above mentioned Marktredwitz Plant in the early nineties was calculated with 600 Deutsche Mark per metric ton, which would be about 300 Euro per ton at the official course at the time of the introduction of the Euro. Thermal desorption maybe today would amount to about 400 Euro per ton, which of course depends very much on the mass of the treated batch. Flotation enhanced soil washing as a solely technology would be easier to handle and considerably cheaper. The treatment in a fixed installed soil washing center of mercury-contaminated soil up to Hg = 1000 mg/kg would be in a range between 180 and 230 Euro per ton. However, thermal desorption (vacuum distillation) remains the only treatment technology for solid waste with Hg greater than 1000 mg/kg.

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RESERVATIONS

The authors reserve all rights for commercial use of information provided in this report, especially in case of filing application for or grant of patents.

DECLARATION OF CONFLICTING INTERESTS

The authors declare that there is no conflict of interest.

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