



Thiourea leaching of gold from processed municipal solid waste incineration residues

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Abstract

Incineration is one of the key technologies in disposal of municipal waste, which produces municipal solid waste incineration (MSWI) residues with high valuable metal contents. The recycling strategy for the MSWI residues is typically focused on the recovery of scrap metals yielding processed municipal solid waste incineration residues (PIR) as the main byproduct. However, the PIR still contains valuable metals, particularly gold, which cannot be extracted by conventional methods. Here, we evaluated the feasibility of using the 0.5–2.0 mm grain size fraction of PIR containing 28.82 ± 1.62 mg/kg of gold as raw material for a two-stage extraction process. In the first stage the alkalic fine-grained PIR was acidified with a solution of 20% (v/v) of HCl-containing flue gas cleaning liquid that is obtained by the municipal waste incineration plant itself as a waste product. In the second stage we leached the acidified fine-grained PIR by thiourea with Fe^{3+} as an oxidant. Application of the thiourea- Fe^{3+} leaching system resulted in recovery of 16.4 ± 1.56 mg/kg of gold from the fine-grained PIR within 6 h of incubation. Due to high gold market prices, upscaling of the suggested technology can represent a suitable strategy for gold recovery from PIR and other MSWI residues.

Keywords Gold · Leaching · Thiourea · Municipal solid waste incineration residue

Introduction

Municipal solid waste incineration (MSWI) is one of the most common technologies for municipal solid waste (MSW) treatment [1–4]. In 2015–2016, there were 512

MSWI plants operating in Europe. These plants burned a total of 80–93 million tons of MSW per year (ca. 27% of the total annual amount of MSW) [2, 5, 6]. This amount of waste was converted to approximately 20 million tons of MSWI residues through incineration, which is about 20–25% of the weight of the input MSW [3, 6]. Reduction of weight leads to a concentration of scrap metals in the incinerated material [7]. Therefore, MSWI residues are commonly used as a secondary source of ferrous (Fe) and non-ferrous (NFe) metals [3, 8]. The amount of scrap in the MSWI residues reaches 7–15% for the Fe fraction and 1–5% for the NFe fraction [6, 9, 10]. The main amount of scrap is extracted with magnets and eddy current separators [3]. These techniques are efficient for the recovery of metals from MSWI residues with particle sizes larger than 2 mm [11]. However, due to disposal of electronic products, the fine-grained fractions of MSWI might be enriched with valuable elements [12–16]. Nevertheless, the finer particles are often separated from MSWI residues and directly mixed with material left after recycling, forming processed municipal solid waste incineration residues (PIR). One of the metals lost in the fine PIR fraction is gold (Au), which stems from fine electronics, such

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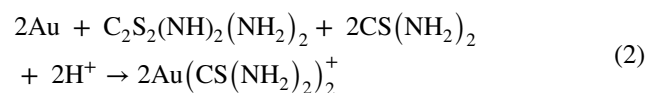
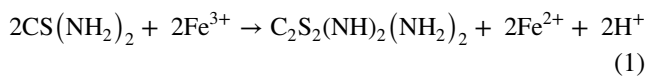
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as soldering of electronic circuit boards [17]. The content of Au in MSWI varies in the range from 0.1 to 21.7 g/ton of MSWI residues [12, 13, 16–21]. This range corresponds to the Au content in the ore of the world's largest Au mines (1.1 g per ton of material on average) [22–24]. Therefore, the MSWI residues can be considered as prospective Au source. However, potentially recoverable separate pieces of Au (e.g., jewelry, coins, etc.) or composite materials (e.g., Au plated stainless steel wristbands of watches) are mainly present in the coarse-grained fraction [21]. The recovery of Au from fine-grained fraction can be performed using traditional pyrometallurgical and hydrometallurgical processes. However, these processes have several limitations. Pyrometallurgical processes (e.g., smelting) require high financial investments for energy and generate hazardous emissions [25]. The density separation cannot be easily adapted for the treatment of MSWI residues due to the presence of reactive substances such as calcium oxide (CaO), metal chlorides, and sulfates. These substances enable hardening and cementation and therefore facilitate mineral incrustation on metal and melt particles that are detrimental to the success of separation [3, 11, 26]. The use of cyanide leaching near populated areas is of widespread concern due to a series of accidents at various gold mines around the world [27].

Steadily rising Au market prices result in a strong motivation for considering the fine-grained fraction of MSWI as a potential source for Au recovery [28, 29]. Although Au is extremely valuable and critical for many industries (e.g., jewelry, electronics, computers, medical, and aerospace), its outstanding chemical resistance becomes a disadvantage in hydrometallurgical processes [30]. The organic compound thiourea ($\text{CS}(\text{NH}_2)_2$) is a promising Au complexing agent [30–32]. Thiourea leaching of Au is typically performed at low pH (1–3) and redox potential (Eh) 400–450 mV (vs SHE) [33]. The process requires an oxidizing agent to form the reactive compound from the thiourea [34, 35]. Ferric ion in sulfuric acid solution can serve as one of the most effective oxidizing agents for Au leaching [36]. Specifically, in acidic solutions, the oxidation of thiourea by Fe^{3+} leads to the formation of formamidine disulfide (Eq. 1). Subsequently, Au is oxidized by formamidine and forms a cationic Au thiourea complex (Eq. 2) [34, 36].



Thus, formamidine acts as an oxidant and as a complexing agent. Application of thiourea for Au extraction from several materials such as domestic ore, chalcopyrite ore, crushed electronic waste, and ceramic wastes allows for the

extraction of up to 90% of Au at optimal conditions (low pH, presence of an oxidant) [34, 37–39]. However, unlike most typically used Au-containing materials, MSWI residues are often characterized by an alkaline pH of 11–13. The increase of pH occurs as a result of hydration of MSWI residues via lime ($\text{Ca}(\text{OH})_2$) formation [40–42]. Therefore, application of the thiourea- Fe^{3+} system for Au leaching requires acidification of MSWI residues [34].

The goal of this study was to test the capability of Au extraction from PIR. Our previous work demonstrated that the 0.5–2.0 mm grain size fraction reached 30.2% of the total MSWI residues weight and contained 28.82 ± 1.62 mg of Au per kg. Furthermore, this fraction was characterized by less buffering capacity compared to the finer fraction (<0.5 mm) and therefore required less acid consumption for neutralization [16]. Thus, we sampled PIR from a regional processing plant, extracted the fine-grained fraction (0.5–2.0 mm grain size) and acidified it with flue gas cleaning liquid produced by the MSWI plant. The produced material was then used for extraction of Au by the thiourea- Fe^{3+} system.

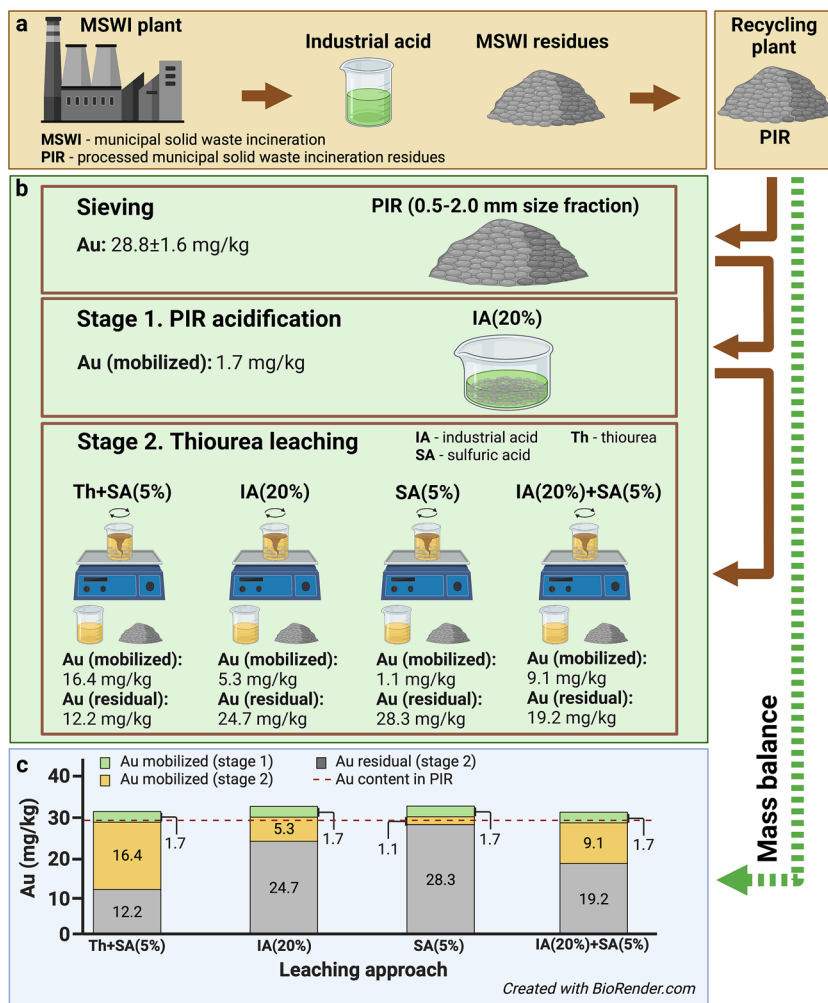
Materials and methods

Source of PIR and industrial acid

The PIR was provided by a recycling plant that processes regional MSWI residues (bottom ash) from the southwestern areas of Germany (States of Hessen and Baden-Württemberg) (Fig. 1a). The plant processes this waste by ageing, sieving, crushing, magnetic recycling and eddy current separation to recover Fe and NFe metal scrap. After recycling, the remaining PIR is either disposed in landfills or used further as construction material. The PIR used in our study had been stored in piles for approximately three months before and approximately three months after the recycling process (from late spring until late autumn) at an open site, exposed to air and precipitation. The PIR consisted of quartz (SiO_2), hematite (Fe_2O_3), calcite (CaCO_3), magnetite (Fe_3O_4), anhydrite (CaSO_4), akermanite ($\text{Ca}_2\text{Mg}(\text{Si}_2\text{O}_7)$) and gehlenite ($\text{Ca}_2\text{Al}_2\text{SiO}_7$) as it was shown in a previous study [16]. Approximately 100 kg of bulk samples were taken from different depths of the pile (1–2 m) and kept in five sealed plastic buckets in the laboratory at room temperature. Before Au leaching, one kg of PIR from each of the five buckets was mixed in a plastic container. The mixed PIR was air-dried for three days, and sieved into fractions of <0.5 mm, 0.5–2.0 mm, and >2 mm with an analytical vibration sieving instrument (Vibratory Sieve Shaker Analysette 3, Fritsch GmbH). The fine-grained fraction (0.5–2.0 mm) was stored at room temperature before acidification and leaching.

The pH value of the PIR was determined using a benchtop pH-meter (inoLab pH 7110, WTW GmbH) equipped with a

Fig. 1 Recovery of Au from PIR. **a** PIR formation. **b** Two-step PIR leaching. **c** Au mobilized from PIR



Mettler-Toledo electrode (InLab Easy DIN). A suspension of 10 g of the PIR in 25 mL of Milli-Q H₂O was shaken manually several times and the pH was directly determined in the settling suspension after one hour, 24 h and one week.

Flue gas cleaning liquid (industrial acid; IA) was provided by the waste-to-energy incineration plant (Fig. 1a). It was produced by purification of flue gas from chlorine in wet scrubbers. The temporal variation in concentrations of the key anions (Cl⁻, SO₄²⁻, Br⁻, I⁻, NO₃⁻ and F⁻) in the IA was monitored by the waste-to-energy incineration plant using ion chromatography (Table 1).

Impact of industrial acid on pH in suspensions of fine-grained PIR

To select the appropriate dilution for the addition of IA to the PIR, we prepared solutions of 5, 10, 20, 25 and 30% (v/v) of IA in Milli-Q H₂O for acidification of the fine-grained size fraction of PIR (0.5–2.0 mm). Duplicate IA solutions (100 mL for each of five dilutions) were used for

acidification of 30 g of PIR (solid:liquid ratio (w:w) ca. 1:3) for 12 days in 200 mL bottles (oxically; non-shaken).

Two-stage Au-leaching process

Stage 1. Acidification of fine-grained PIR

One kg of fine-grained PIR was acidified with 3 L of IA solution (solid:liquid ratio (w:w) 1:3) diluted in Milli-Q H₂O

Table 1 Content of key constituents in the flue gas cleaning liquid (industrial acid)

Constituent	Concentration range	Unit
Cl ⁻	80–130	g/L
SO ₄ ²⁻	0.5–6.0	g/L
Br ⁻	0.1–2.8	g/L
F ⁻	0.4–1.5	g/L
I ⁻	3–43	mg/L
NO ₃ ⁻	15–450	mg/L

(20% v/v) (Fig. 1b). The treatment was done in duplicates in standing mode (non-shaken) in two plastic containers at room temperature. During incubation, we followed pH and sampled (in triplicates) the overlying IA solution for Au quantification hourly. To quantify the Au content, 3 mL of the solution was transferred into 15 mL centrifuge tubes and centrifuged for 10 min (7000 g). The supernatant was filtered with 0.2 µm syringe filters (Puradisc FP 30 CA, Whatman) and diluted in 10% Aqua Regia solution to maintain Au in the dissolved state. All samples were kept at 4 °C before Au quantification with inductively coupled plasma—optical emission spectrometry (ICP-OES). After 24 h of incubation, the IA solution was decanted completely. The remaining acidified PIR was centrifuged for 15 min (7000 g) to remove excess liquid. The PIR was dried at room temperature for 4 days and stored for further processing.

Stage 2. Mobilization of Au from fine-grained PIR with thiourea

After air-drying, 100 g of the fine-grained acid-treated PIR was transferred into eight glass beakers with magnetic stir bars (Fig. 1b). The acid-treated PIR was extracted with one of the following leaching solutions (Milli-Q H₂O): i) 300 mL of 5% sulfuric acid (SA; v/v) containing 20 g/L thiourea and 10 g/L Fe₂(SO₄)₃·7H₂O; ii) 300 mL of 20% IA (v/v); iii) 300 mL of 5% SA (v/v); or iv) 300 mL of a mixture of 20% IA and 5% SA (v/v). The incubation of all setups was performed in duplicates each for six hours at room temperature under stirring (300 rpm). Samples for pH and Eh monitoring as well as leachate samples for Au quantification were taken hourly following the same protocol as described above. Eh was determined using a pH-meter (Mettler-Toledo; SG2) with Pt and Ag/AgCl electrodes.

The pregnant leaching solutions were removed from each of the eight beakers, the residual PIR was collected, centrifuged for 15 min (7000 g) to remove excess liquid, dried at room temperature for four days, and used for Au quantification following the protocol described below. All solid-phase samples were stored in a desiccator with silica gel at room temperature for further digestion and subsequent Au quantification.

Elemental analysis

Triplicates of air-dried samples of the fine-grained PIR as well as samples of leaching residuals were milled to a fine powder with a planetary mill (Fritsch Pulverisette, Fritsch GmbH). To minimize the risk of metal contamination, the milling buckets were cleaned with pure sand in a 10 min run (at grade 5–6), followed by an additional 10 min run with sample material to prime the milling buckets. The samples

were then milled for 25 min. After milling, the powdered samples were dried at 105 °C for 48 h and stored in 50 mL polypropylene centrifuge tubes (Orange Scientific or SLG Süd-Laborbedarf Gauting) at room temperature in a desiccator dryer with silica gel.

Closed microwave digestion of powdered samples was performed in a microwave system (Multiwave Go Microwave Digestion System, Anton Paar). To this end, 0.25 g of samples were weighed in modified polytetrafluoroethylene (PTFE-TFM) digestion vessel and extracted with 0.5 mL of Milli-Q H₂O, 3.6 mL of 37% HCl, and 1.2 mL of 65% HNO₃. All samples were extracted in triplicates. The following temperature gradient parameters were applied in the microwave digestion process: step 1 (10 min, 180 °C), step 2 (20 min 200 °C), step 3 (30 min, 180 °C), ventilation (> 30 min). After cooling, the samples were subsequently filtered (filter paper, 619 G ¼, Macherey–Nagel) into 100 mL measuring flasks. Digestion vessels and the residues were rinsed several times with Milli-Q H₂O to a final volume of 100 mL. Aqua regia digests were stored in polyethylene bottles at 4 °C until further analysis. Before analysis samples were filtered with 0.2 µm syringe filters (Puradisc FP 30 CA, Whatman). ICP-OES (SPECTROBLUE TI, Ametek) was used for quantification of Au in PIR digests as well as in diluted leachate samples (Online Resource 1).

Materials and solutions

All plasticware and glassware that were used for analytical work were pre-cleaned with 1 M HCl overnight and rinsed with Milli-Q H₂O. The following reagents were used for leaching experiments and for subsequent analysis: 25% H₂SO₄ (for analysis; Merck); thiourea (99%, extra pure; Acros Organics); Fe₂(SO₄)₃·7H₂O (puris p.a. Sigma-Aldrich); 65% HNO₃ (for analysis; Merck); 37% HCl (puris p.a.; Merck); Milli-Q H₂O (18.2 MΩ·cm; Millipore); noble metals multi-element standard (VHG labs).

Results and discussion

Gold content in the fine-grained PIR size fraction

The PIR's composition was heterogeneous even after recycling, as it still contained large pieces of scrap and NFe metals. The fine-grained size fraction (0.5–2.0 mm) of PIR was separated and weighed. It constituted 30.2% of the total PIR weight. In our previous study we demonstrated that the fine-grained fraction contained 28.82 ± 1.62 mg of Au per kg of PIR compared to an Au content of ca. 21.69 mg per kg in the raw PIR material [16]. Therefore, the 0.5–2.0 mm size fraction was selected to investigate Au extraction from PIR.

Acidification of fine-grained PIR size fraction with industrial acid

Storage of PIR in open-air waste piles contributes to weathering and capturing atmospheric CO₂ which lowers the pH of the material via calcite (CaCO₃) and ettringite (Ca₆Al₁₂(SO₄)₃(OH)₁₂ × 26H₂O) formation [41, 43–45]. However, the six-month storage period of the PIR did not lead to an appreciable pH decrease. The pH of the fine-grained fraction remained high (10.5 ± 0.5), indicating the presence of hydrated lime. Since the leaching efficiency of the thiourea-Fe³⁺ solution is determined by the formation of anionic complexes that dissolve Au, a strong ability of the leaching solution to decrease the pH value of the PIR is essential [38]. This can be achieved by treatment of PIR with acidic solutions, for example with industrial acid. Typically, the IA is disposed after neutralization and reduction of the heavy metal load [46]. However, due to the high HCl content (8–13% v/v; Table 1) it can be used instead for acidification of the fine-grained PIR. Therefore, we tested the capability of IA solutions (5, 10, 20, 25 and 30%; v/v) to decrease the pH of PIR suspension. Low pH values allow Fe³⁺ to oxidize thiourea accompanied by the formation of formamidine disulfide (Eq. 1), which in turn oxidizes Au forming a cationic Au thiourea complex [34, 36]. Triplicate IA solutions were mixed with fine-grained PIR and incubated oxically for 12 days. The pH of all PIR suspensions increased sharply during the first day of incubation (Fig. 2). However, the pH of the PIR suspensions containing 10, 20, 25 and 30% of IA (v/v) remained low and sufficient for oxidation thiourea by Fe³⁺. After 12 days of incubation, the pH values of the 5 and 10% solutions increased above 7 and 5, respectively, while the pH values of the 20, 25 and 30% solutions stabilized at a range of 2.9 to 3.8. These results demonstrated that treatment with 20–30% IA was sufficient to maintain pH values

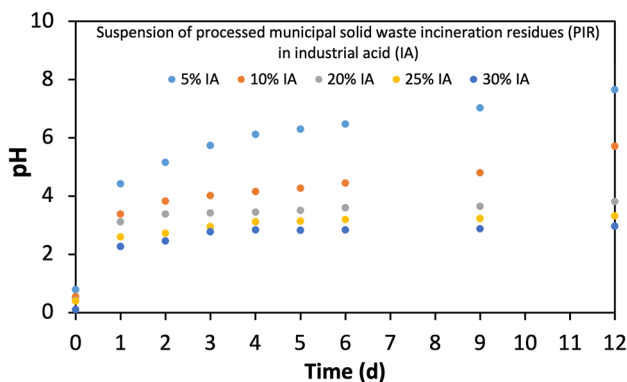


Fig. 2 pH dynamics in suspensions of PIR. PIR was resuspended in 5, 10, 20, 25 or 30% IA. Markers represent average of duplicate pH values (Online Resource 2)

below 4 as required for effective Au extraction using Fe³⁺ as an oxidant [34, 36].

Based on these results we further acidified 1 kg of fine-grained PIR material with 3 L of 20% IA solution (Fig. 1b). Temporal pH monitoring of PIR suspension demonstrated that after six hours of incubation, the pH continued to increase but only slightly (Fig. 3). At this time, a sufficient portion of salts could be dissolved thereby reducing the alkaline capacity of PIR. The effect of PIR acidification on Au mobilization was also determined (Fig. 3). Quantification of dissolved Au demonstrated that 1.74 ± 0.4 mg of Au per 1 kg of fine-grained PIR were mobilized. This amount of Au was equivalent to 5.9 ± 0.5% of the total Au content in the fine-grained PIR (28.82 mg/kg) [16]. The mobilization of Au by 20% IA solution was supposedly caused by the peculiarities of IA composition (Table 1). Specifically, the HCl and HNO₃ could partially digest and dissolve the bulk PIR material, while lixivalents (e.g., bromide and chloride) could potentially have complexed and mobilized the Au [30, 31, 47]. Chloride and bromide are typically applied together for Au leaching. Thus, several studies demonstrated earlier that Au can be complexed by chloride (120–210 g/L) and bromide (23.7–102.7 g/L) at atmospheric pressure [48, 49]. The 20% IA solution contained lower concentrations of both chloride (16–23 g/L) and bromide (0.2–0.56 g/L). However, they could still partially mobilize Au, while concentrations of iodide was not sufficient for complexation of Au [50, 51].

Leaching of Au from acid-treated fine-grained PIR using the thiourea-Fe³⁺ extraction system

At the second step of Au recovery, 300 mg of fine-grained acid-treated PIR was resuspended in 100 mL of thiourea in 5% sulfuric acid (SA) with Fe³⁺ as an oxidant. Leaching was accompanied by mixing of the PIR suspension

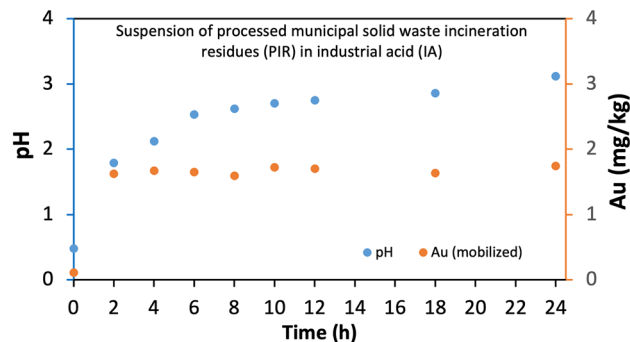


Fig. 3 pH dynamics and Au content in suspension of PIR in 20% IA. Markers represent average of duplicate pH and Au concentration values (Online Resource 3)

to improve extraction efficiency. Furthermore, to reveal the contribution of Au complexation by thiourea, we additionally incubated the fine-grained fraction of the PIR in 20% IA solution without the thiourea- Fe^{3+} system. Additionally, to monitor the impact of low pH value on Au recovery, the fine-grained fraction of the PIR was incubated in 5% SA solution, and in a solution of 20% IA and 5% SA (Fig. 1b). The pH and Eh values during incubation were clearly dependent on the concentration of acids in the leaching solution. Thus, the incubation of fine-grained PIR in the solution containing 20% IA and 5% SA for six hours led to a minor change of the pH and Eh values from 0.01 to 0.27 and from +406 to +387 mV, respectively (Fig. 4a, b). Incubation of fine-grained PIR material in 20% IA resulted in more noticeable changes of pH and Eh. During six hours of incubation, the Eh decreased from +395 to +256 mV while the pH increased from 0.12 to 2.51. The application of thiourea in 5% SA maintained the pH and Eh values comparatively stable during incubation, i.e., the Eh value decreased slightly from +393 to +344 mV while the pH increased from 0.24 to 0.99. Similar values were observed for incubation in

5% SA, where the pH value increased from 0.20 to 0.87 while the Eh value decreased from +398 to +369 mV. In general, these results correlated well with the total acid content where the values showed the largest changes with decreasing acid strength, i.e., IA (20%) + SA (5%) < thiourea + SA (5%) = SA (5%) < IA (20%), and thus the highest acid content maintained the lowest pH and highest Eh values.

Ferric iron oxidizes thiourea and leads to the formation of formamidine disulfide (Eq. 1), which in turn oxidizes Au and forms a cationic Au thiourea complex (Eq. 2) within a Eh range from +350 mV to 450 mV (vs SHE). However, treatment of acidified fine-grained PIR with IA (20%) + SA (5%), thiourea + SA (5%) and SA (5%) maintained an Eh value from +340 mV to +390 mV (from +539 mV to +589 mV; vs SHE). Higher (more positive) redox potentials can lead to undesirable oxidative degradation of thiourea via formamidine disulfide ($\text{NH}_2(\text{NH})\text{CSSC}(\text{NH})\text{NH}_2$) formation, which can decompose into thiourea ($\text{CS}(\text{NH}_2)_2$), cyanamide (NH_2CN) and elemental sulfur (S°), which covers the Au particles and prevents its mobilization [33]. Nevertheless, the application of thiourea in 5% SA led to a mobilization of 15.1 ± 0.7 mg of Au per kg of fine-grained PIR during the first two hours of leaching (5.02 ± 0.22 mg/L of Au in the leaching solution). After six hours of leaching, the Au concentration reached 5.46 ± 0.52 mg/L, corresponding to 16.4 ± 1.56 mg Au leached per kg of fine-grained PIR material ($56.8 \pm 5.4\%$ of the total Au content; Fig. 3c). Under controlled leaching conditions, the solution of 20% IA contributed to the release of 5.3 ± 0.2 mg/kg of Au ($18.4 \pm 0.5\%$ of the total Au content; Fig. 4c), while the application of a mixture of 20% IA and 5% SA led to the release of 9.1 ± 0.1 mg/kg of Au ($31.5 \pm 0.2\%$ of the total Au content). These results were supposedly achieved due to digestion/dissolution of the PIR bulk material by the HCl acid and complexation of the released Au by the lixivants (chloride and bromide) that were present in the IA (Table 1) [30, 31, 47]. Finally, the incubation of fine-grained PIR in 5% SA resulted in a release of 1.1 ± 0.9 mg/kg of Au ($4.0 \pm 3.1\%$ of the total Au content) supposedly due to dissolution of chlorides, nitrates and other salts from the PIR bulk phase followed by Au complexation. The concentration of chloride in municipal waste incineration residues can reach 1–3 wt% [52, 53] and most of the chloride in the MSWI residues is present in the fine-grained fraction [54]. The total Au contents consisting of mobilized Au and Au left in the PIR in all treatments were equivalent to 30–32 mg/kg (Fig. 1c). These concentrations were comparable to the concentration of Au determined in the original untreated fine-grained PIR (28.82 ± 1.62 mg/kg) [16]. Therefore, the application of thiourea- Fe^{3+} system as well as other leaching agents did not lead to a complete Au extraction from PIR and more than 40% of the Au was still left in the remaining PIR material.

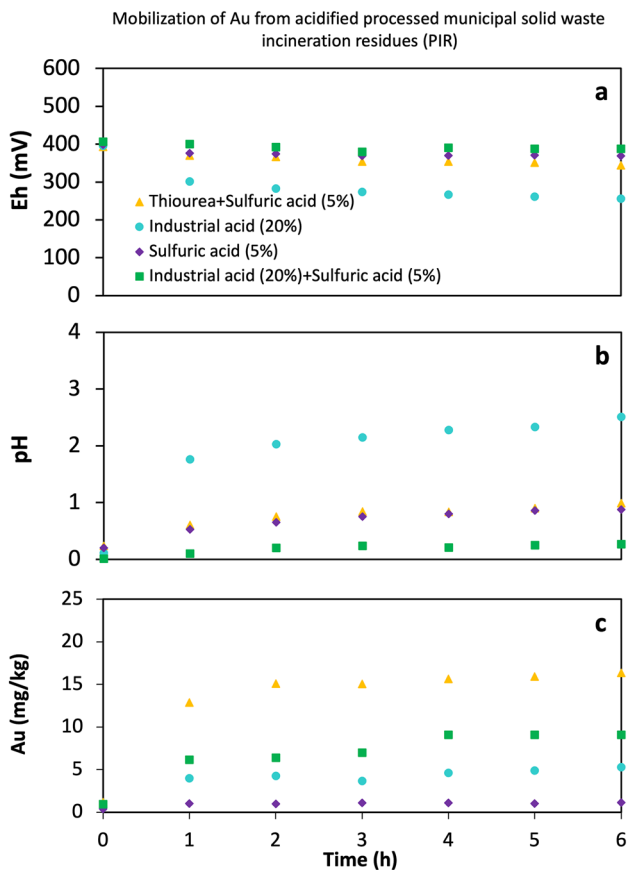


Fig. 4 Eh (a), pH (b) and Au content (c) in PIR suspensions. Markers represent average of duplicate values (Online Resources 4–6)

Potential techniques of Au recovery from thiourea-Fe³⁺ leaching solution

Our results have shown that the efficiency of Au mobilization by the thiourea-Fe³⁺ system from acid-treated fine-grained PIR was the highest among the tested treatments. The next step after the mobilization of Au is its recovery from the pregnant leaching solution. However, the techniques of Au recovery from dissolved state are not well-established so far. Conventional methods such as the Merrill-Crowe process wherein Au is recovered from solution by cementation with zinc powder or methods that involve the use of activated carbon followed by electrolysis were developed for the Au cyanidation approach [39, 55–58]. Several studies demonstrated that Au can be recovered from thiourea leaching solution by adsorption on activated carbon [59–61] and organic materials (e.g., persimmon tannin based sorbents, rice husk or chitin) [61–64] or via ion exchange [65, 66]. The adsorption capacities of activated carbon, rice husk and chitin are relatively high and allow to recover ca. 36 mg/g, 28 mg/g and 58 mg/g of Au, respectively, from the thiourea leaching solution [61, 64]. These methods might be used for recovery of Au from leachate produced by treatment of PIR, since the application of IA for acidification of PIR can also reduce the amount of soluble metals (particularly Fe and Cu) thereby reducing the competing adsorption.

The selective recovery of Au from thiourea leaching solution might be achieved by electrowinning [67]. However, the efficient electrowinning requires leachate with high Au concentration [67, 68]. This requirement, therefore, does not allow considering electrowinning for Au extraction from PIR leachate. The precipitation of Au from thiourea solution by H₂ reduction is another method that has been successfully applied. However, this process is carried out at high pressure and temperature and requires the application of a catalyst [69]. Nevertheless, the precipitation can be also considered as a potential method for recovery of Au from PIR leachate. For example, the precipitation of Au by sodium borohydride in acidic solution of thiourea may facilitate the selective reduction of Au and even allows to recycle the thiourea [70]. Finally, cementation can be used for recovery of Au. Several metal species have been shown to be useful cementing agents for Au from thiourea solutions including Cu, Zn, Ni and Al [71–73]. However, the Fe³⁺ used as oxidizing agent for leaching of Au from PIR can significantly affect the cementation reaction kinetics via increasing of redox potential of the solution. Nevertheless, the high redox potential can be lowered by supplementation of the cementation reaction with sodium citrate which can form Fe³⁺-citrate complexes [73].

In summary, the recovery of Au from the thiourea complex might be performed by different techniques (e.g., adsorption, precipitation, cementation, and electrowinning).

However, low Au and high Fe³⁺ contents of the PIR leachate may decrease the efficiency of the extraction process. Therefore, the acidification of PIR with IA can help to decrease concentration of metals that can be mobilized during thiourea-Fe³⁺ leaching of Au. In turn, the lower concentration of dissolved metals (primarily Fe and Cu) can potentially increase the efficiency of Au extraction by adsorption, via reducing the competing adsorption, or by cementation via reducing the redox potential of the solution. In addition, the application of sodium citrate may help to further neutralize the effect of residual Fe³⁺ on Au recovery from the leaching solution.

Use of secondary materials for Au leaching

Approximately 2551 tons of Au (63.44% of global Au demand) were used for production of jewelry and electronics in 2021 [74]. Increasing production of Au-containing goods will inevitably lead to increased amounts of Au in the various types of waste (e.g., sweeping jewelry, electronic wastes, slags, etc.) [12, 13, 25, 75–82]. Therefore, over the past decades there have been several attempts to recover Au from secondary materials through leaching techniques using aqua regia, cyanide, ionic liquids, thiosulfate, or thiourea as lixiviants [27, 30, 68]. Among these commonly used lixiviants, thiourea is relatively effective in Au mobilization whilst having the least negative impacts on the environment [68, 83, 84]. Indeed, the application of different thiourea-based leaching systems is usually accompanied by a high yield of mobilized Au. Thus more than 80% of Au can be mobilized by thiourea leaching from such wastes as activated carbon (2.9 µg/g of Au) [85], wastes of amalgamation and cyaniding processes (1.7–8.5 µg/g of Au) [86], and Cu smelting slag (0.44–0.46 µg/g of Au) [87]. Furthermore, thiourea-based leaching systems are often used for extraction of Au from electronic wastes [14, 68, 76, 83, 84, 88, 89]. For example, more than 90% of Au can be leached from dust generated during processing of electronic waste (141 µg/g of Au) or printed circuit boards (PCBs) (43 µg/g of Au) [88, 89].

The high efficiency of Au recovery by thiourea-based leaching often requires the pretreatment of secondary materials. In particularly, the size reduction [62, 90], the removal of impurities (plastics, metal peace, etc.) [91, 92] or pre-leaching of other metals included in the composition of the Au-containing secondary material [93] help to increase the efficiency of the following Au-leaching process. Pre-leaching of Fe and Cu is especially important for Au mobilization by thiourea since the dissolution of these metals increases the redox potential of acidic leachate thereby oxidizing thiourea and forming formamidine disulfide [90, 94–96]. Different inorganic acids HCl, HNO₃ and H₂SO₄ with or without additional oxidants (e.g., O₂, H₂O₂), organic acids (e.g., citric acid, oxalic acid and acetic acid), and other

lixiviants (e.g., EDTA, sodium acetate and sodium hydroxide) can be used for the recovery of Fe and Cu from Au-containing secondary materials [68, 97, 98]. These methods have consistently demonstrated a high efficiency of Fe and Cu leaching and recovery. For example, fly ash from waste incineration plants (L/S ratio—20) that was treated with 5–7 M HCl extracted ca. 80% Fe and 86% of Cu [97]. The ammonia (5 M)/ammonium sulphate (2 M) leaching system supplemented by oxidants (O_2 or H_2O_2) can extract up to 91% of Cu from PCBs (L/S ratio—20) [99]. EDTA (4 M / 1 M of total heavy metals content) can facilitate the extraction of Cu up to 88% from electrostatic precipitator ash from an incineration plant (L/S ratio—10) [100], while 0.02 M ammonium citrate promoted the mobilization of Cu and Fe from MSWI bottom ash (L/S ratio—10) [101]. Copper can also be volatilized from the MSW fly ash at 700–900 °C in the presence of poly(vinyl chloride). This method enables the recovery of ca. 50% of Cu [4]. In addition, the efficiency of Fe and Cu recovery can be improved with the application of microwave radiation [87]. Bioleaching can be also considered as a promising method for pretreatment of secondary materials. Thus, *Acidithiobacillus ferrooxidans* in association with *A. thiooxidans* [102] or alone [103] can be used for leaching of Cu from PCBs. In both cases, the efficiency of Cu recovery exceeds 90%.

When considering the existing applications of thiourea-based leaching as well as the rising price of Au (ca. 57,454 USD per 1 kg in January 2020) [104], Au recovery from fine-grained PIR using the suggested two-step thiourea- Fe^{3+} extraction procedure could represent a new high-profit niche for the MSWI residue recycling industry. The high content of Au in the PIR (28.82 ± 1.62 mg/kg) and the high extraction efficiency (more than 50%) allows us to consider the fine-grained PIR fraction as a valuable urban ore comparable in Au content with other types of secondary materials [27, 105, 106]. Upscaling of laboratory technology is a complex process that can lead to losses in Au-leaching efficiency. However, if the efficiency of Au extraction remains at the level of 50% thiourea- Fe^{3+} , extraction procedures will mobilize up to 14 g of Au per 1 ton of PIR. This amount of Au is equivalent to 804.5 USD according to the current price [104]. Further research is needed to assess the operating costs of this technology.

Conclusions

The sufficient part of MSW (ca. 27% in Europe) is currently burned in incineration plants [2, 5, 6]. The recovery of valuable metals (e.g., Au) from MSWI residues (e.g., PIR) by conventional methods including ageing, sieving, crushing, magnetic separation, density separation and eddy current separation have several limitations due to the relatively low

abundance of precious metals and the high structural and compositional complexity of the incineration waste matrix [25]. Therefore, there is a strong need to develop and apply low-cost and eco-friendly methods to recover precious metals. This study presents a two-stage leaching process for Au from fine-grained PIR material. Although this fine-grained fraction was characterized as a highly alkaline material, it was efficiently acidified by a pretreatment with 20% IA, resulting in the extraction of up to 6% of the total Au content. The second extraction step, using a thiourea- Fe^{3+} leaching system, led to the recovery of a large fraction of Au from PIR material (in total $56.8 \pm 5.41\%$ of total Au content) during only six hours of incubation. Due to the high Au price and the rising need for Au in electronics, we suggest that upscaling of the thiourea- Fe^{3+} leaching approach can be applied for treatment of the fine-grained fraction of PIR and Au extraction. Furthermore, this approach has the potential to be not only cost-effective but also environmentally friendly in comparison with cyanide leaching, which is highly toxic. Therefore, we can assume that recovery of Au with thiourea- Fe^{3+} system has the potential to become an integral part of sustainable waste management in the future.

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Declarations

Conflict of interest None.

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