

# Cyanide alternative leaching reagents for gold recovery from electronic waste: Potential and limitations of thiosulfate

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#### **Abstract**

Gold has excellent chemical resistance and electrical conductivity. These featured properties make it a useful material especially in the electronical industry. Golds rareness and wide field of applications reason the high price and give an economical motivation for recycling. The outstanding chemical resistance of gold becomes a disadvantage when it comes to gold metallurgical. Applied processes like mercury, chloride and mainly cyanide are mostly aggressive and very toxic. Due to ecological impacts, industry and researchers worldwide are looking for efficient alternative gold recovery methods. For this, different less or non- toxic cyanide alternative leaching reagents came up as promising alternatives in the last decades. These reagents mainly were tested on gold ores, but are also suitable for electronic waste, which shows a significantly higher gold content.

In this study, promising various leaching reagents are discussed and compared at the beginning. Based on this the most promising reagents for gold recovery from electronic waste was determined: thiosulfate. The reagent was tested on different gold containing e-waste samples like flue dust from crushers, comminuted and untreated boards. Thiosulfate did not show satisfying results for flue dust and comminuted boards, owing to the excessive reagent consumption by metallic impurities. It was found that an efficient gold recovery via thiosulfate leaching requires a strict process control, which is hardly manageable when a complex input material like crushed e-waste is used. In superficial leaching experiments of holistic RAM's a formation of a passive layer was observed, which hindered or slowed down the gold dissolution. This effect can be explained due to oxidative degradation of thiosulfate by copper complexes and dissolved oxygen. Further experiments aimed on the stabilization of TS in solution revealed a gold recovery of up to 98.9 % gold within 48 hours (0.15 M ammonium thiosulfate, 0.6 M ammonia, 0.02 M Cu(II)sulfate, 30 °C, 300 rpm). Due to the low oxidative conditions,



time is the most important parameter at this solution composition for gold recovery and selectivity in terms of an occurring co-extraction of Cu, Ni and other elements.

#### Introduction

Since its inception in the late 1800s, cyanidation is the dominant process for the recovery of gold and silver from ores. Due to its simplicity and high cost efficiency, it replaced former processes nearly completely. Nowadays, the cyanide process accounts for more than 90 % of primary gold extracted in the world. The main disadvantage of cyanide is related to environmental issues, which may occur by improper storage and transport or failed tailings management and storage. Cyanide is highly toxic to humans and living beings even at very low concentrations. Another disadvantage of cyanide is its low dissolution rate, compared to many alternative leaching reagents. In order to overcome environmental problems and technical disadvantages, new leaching reagents were increasingly researched in the past decades [1-7].

With an annual growth of around 5 %, e-waste is one of the fastest growing waste streams in the world. In 2018 approximately 49.8 million tons arose worldwide, so that e-waste accounts for approximately 5 % of the municipal solid waste. Its complex composition and the fact that more than 60 elements of the periodic table are used in electronics justify the challenging recycling process. Bulk metals are copper, aluminum and iron, which make up to more than 30 wt.-%. Further valuable elements like precious metals show a concentration of less than 0.05 wt.-% in total. Besides metals, main components are plastics, ceramics and glass fibers but also may be wood, cloth or other. The total material value of e-waste was estimated to be 55 billion € in 2016 (Table 1). Despite their low concentration, noble metals account for a disproportionate value share [8 – 11].

Table 1: Emerged e-waste, corresponding value share (left) and average composition (right) of major materials in 2016 [10]

	Mass [kt]	Value [Mio €]	Composition
Fe	16,283	3,582	
Plastics	12,23	15,043	Ag, Au, other;
Al	2,472	9,585	Pd; 26.1%
Cu	2,164	9,524	0.05% Fe; 36.3%
Ag	1.6	884	Cu; 4.8%
Au	0.5	18,840	AI; 5.5%
Pd	0.2	3,369	Plastics; 27.3%



The state of the art processing of e-waste consists of mechanical-physical pretreatment with the aim to enrich the valuable metal concentration and a metallurgical recovery in the copper route. Precious metals are collected in the copper phase and pass different refining steps until they are separated via electrochemical deposition. In subsequent steps the precious metals are further enriched and recovered separately. This process shows some significant drawbacks, such as an expensive pretreatment, gold loss due to dissipation, the need for a variety of time consumptive processing steps until precious metals can be recovered [12, 13].

Although cyanide is the established gold recovery process, there are many reagents which are also capable to form soluble gold complexes. But only thiosulfate, thiourea, halide, thiocyanate and sulfide can be identified as technically and economically favorable alternatives to cyanide [1, 2]. Figure 1 shows typical leaching conditions for relevant lixiviants. Besides a complexing agent, also an oxidizer is necessary to transform gold from its metallic state into a soluble compound. However, oxidizing conditions can also cause a decomposition of reagents, in particular for thiosulfate and thiourea. Acidic leach systems tend to operate at high reagent concentrations and temperatures, can lead to corrosion problems. The advantage of alkaline systems lies in a higher selectivity, which is important for complex multi-metal systems like e-waste. Furthermore, the subsequent treatment of process residues from alkaline systems is less expensive and corrosion of leaching tanks is neglectable. Reagents like thiosulfate and iodide show high environmental safety, compared to cyanide. The leaching rate of the reagents was reported to show following order: halides > thiourea > thiosulfate > cyanide [2, 13 – 18].

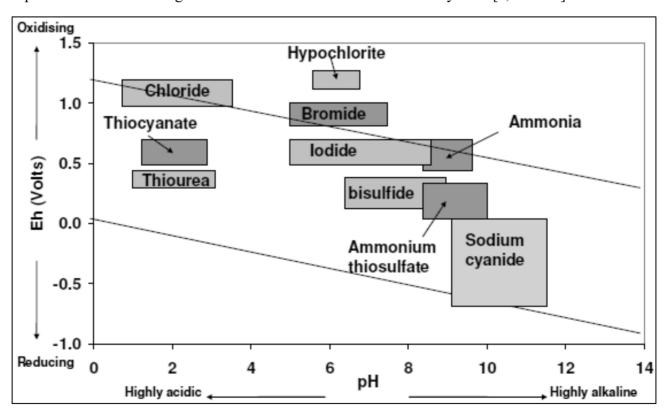


Figure 1: Eh-pH diagram depicting typical leaching conditions for various complexing agents [14]



Cyanide alternatives that have received particular recognition in the last decades are thiosulfate, thiourea, halide, thiocyanate and sulfide systems [19]. Table 2 presents comparative features of relevant gold lixiviants with regard to technical properties, economics, applicability and toxicity.

Table 2: Comparison and evaluation of gold lixiviants for e-waste [3, 4, 20 - 22]

	Leaching rate	Process robustness	Reagent costs	Reagent loss	Selectivity	Toxicity	Research level
Cyanide	-	+	0	+	-	-	+
Thiocyanate	0	+	0	+	0	0	-
Thiourea	0	0	0	-	-	-	+
Bisulfide	0	0	-	0	-	0	0
Halides	+	+	-	0	+	- / o	
Thiosulfate	0	-	+	-	+	+	+

Although thiosulfate shows some drawbacks like a high reagent consumption and a complex dissolution reaction, it is the most promising leaching reagent for the hydrometallurgical gold recovery from e-waste. Main advantages of thiosulfate are its high environmental compatibility, the low solubility for ignoble metals, very low reagent prices and reasonable leaching rates for gold [22 - 29].

# **Experimental**

Aim of the experimental investigation is the identification of suitable leaching parameters to enable an efficient gold recovery from e-waste. For this purpose, thiosulfate (TS) was selected as most promising reagent in the aforegoing comparative assessment. The pre-treatment of the input material represents an important parameter and requires special attention. Due comminution besides gold from integrated circuits also a variety of different other metals and materials are liberated and occur on the surface, i.e. they may come into contact with the leaching solution. Further parameters like leaching of different e-waste types (Figure 2), solution composition (in particular TS, ammonia and copper concentration), are investigated along with temperature, leaching duration, pH and oxygen concentration. Besides gold, the co-extraction of further valuable metals via thiosulfate leaching will be observed and evaluated.



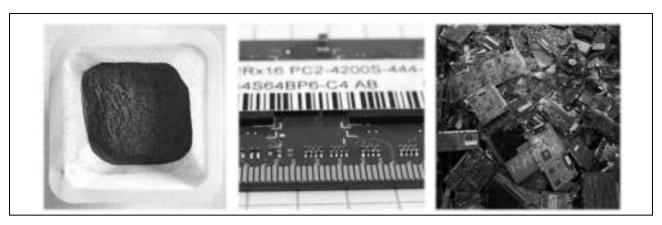


Figure 2: Samples used for experiments on TS gold leaching, from left to right: flue dust from e-waste crushing and milling processes, RAM and circuit boards

Table 3: Flue dust composition in wt.-%

	Au [ppm]	Ag [ppm]	Cu	Al	Fe	Ni	Zn	Pb	Sn	Si	Other <sup>1</sup>
Flue dust	29	580	9.83	12.49	1.83	0.09	3.49	1.88	0.92	7.95	56.54
RAM stick	211	800	4.19	4.75	0.14	0.42	0.12	0.01	4.53	28.38	47.64
Printed boards	191	732	5.54	8.29	0.31	0.09	0.17	1.95	2.77	13.47	51.99
<sup>1</sup> mainly C. O. F									С. О. Н		

The leaching experiments took place on stirred heating plates (SLR, SI Analtics). Further relevant solution properties like pH level and oxygen concentration (mg/L) were measured with a WTW ph1970i and WTW inoLab Oxi 7310. Thiosulfate and other chemicals (reagent grade or higher) were acquired from AlfaAesar and SigmaAldrich. Deionized water was used from a mixed bed water deionizer (TKA DI 2800 or similar). Solid samples were analyzed via XRF (Panalytical Axios 2005), while the composition of liquid samples was determined through inductively coupled plasma optical emission spectroscopy (ICP-OES, Spectro Arcos 2013). Due to the low stability of thiosulfate solutions, leaching residues were digested with aqua regia (AR) for chemical analysis. Surface analysis, microscopy scanning electron microscopy (SEM) and electron dispersive spectroscopy (EDS) took place with a FEI SEM (Quanta-650F SEM, Thermo Fisher Scientific). For sample weighting a special accuracy weighing machine was used (XS603S DeltaRange, Mettler Toledo).

Different experimental approaches were defined to identify suitable process conditions for an efficient gold recovery (Figure 3). The direct leaching takes advantage of the design of electronic components. Just a very limited number of metals are present on the surface of electronic components, i.e. solders and electrical. This enables a high potential for the superficial metal extraction. Inert layers of epoxy resin on the surface cover other materials and methods, which do not take part in the leaching process. Main disadvantage of this process is the lacking possibility to recover gold which is used in integrated circuits (CPU and RAM) or other components which cover gold with inert materials. An aforegoing treatment like comminution and thermal conditioning aims on the utilization of different methods to separate disturbing elements, liberate valuable metals and enrich the metal fraction. Gold from CPU and similar components could be liberated by this treatment. Besides different mechanical-



physical methods also thermal conditioning shows significant advantages in the processing of e-waste and therefore are tested on their applicability for gold recovery via TS leaching. The thermal conditioning of e-waste enables the decomposition of organic compounds to produce a solid product with enriched metal content [30, 31].

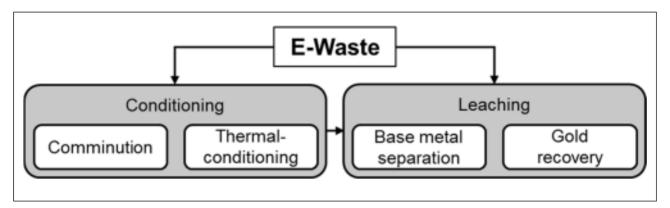


Figure 3: Illustration of investigated e-waste pre-treatment procedures

#### Results and Discussion

#### Thiosulfate Leaching of Flue Dust

Flue dust arises in e-waste shredding processes and contains the same elements as e-waste in general but in a different allocation. The low gold concentration enables an important demonstration of the selectivity of TS [32]. Investigated parameters were among others: concentration of TS, ammonia and copper-sulfate, leaching duration, injection of air and oxygen, addition of additives. Resulting from 40 experiments at different parameters, best experiments just yielded in a recovery of 15.2 % to 23.3 % within 6 h by injection of air and oxygen (Figure 4). At the same time up to 39.9 % Ag and 61.8 % Cu were dissolved, causing a reagent consumption of more than 0.12 mol in a 0.1 M TS solution. There was no measurable dissolution of Fe, Al, Sn, Pb. The positive effect of aerated solutions on gold dissolution is due to better oxidative conditions, as it was also observed by Molleman et al. [33]. A test series with varied leaching durations (3, 9, 24, 48 h) revealed an interesting development of gold recovery. After 3 h the highest gold recovery (31.4 %) was achieved, decreasing with time to 23.3 % after 24 h and 15.2 % after 48 h. This trend indicates a complete reagent consumption already after 3 hours or less. The decreasing recovery can be explained with the precipitation of gold via cementation on the surface of less noble metals, which form a more stable TS compound [28, 32, 34, 35].



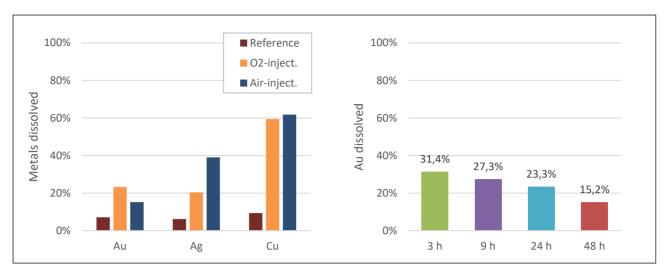


Figure 4: Results of flue dust leaching experiments (0.1 M TS, 0.4 M NH<sub>3</sub>, 10 mM Cu, 25 °C, t = 6 h, s/l = 1/20)

The low gold concentration itself does not justify its insufficient recovery. In different experimental work on thiosulfate from literature it was found that a yield of 89 % can be achieved using ore type samples with 32 ppm gold, where the sulfidic ore consists of around 60 wt.-% iron 127 ppm copper and 12 ppm silver, mainly as chemical compound. Thus, the potential reagent consumption in this leaching system is strictly reduced due to a lower Cu and Ag concentration, unlike it is the case for secondary resources [36]. Similar experiments proved a higher selectivity for copper and silver containing ores than with cyanide [37, 38].

# **Indirect Gold Recovery**

The thermal treatment of PCB'S enabled a holistic decomposition of organic compounds and an accompanying mass reduction of around 25 %. The subsequent comminution and sieving steps enabled a further enrichment of gold. The comminution of e-waste is significantly facilitated by an aforegoing decomposition of soft and deformable organic compounds. Ductile metals like Cu, Al and Fe can be separated in a coarser sieve fractions of > 90  $\mu$ m (Figure 5). The total weight reduction of PCB's after thermal pre-treatment and sieving makes up 35 – 65 %. The gold content can be enriched by 1.32 to 2.39 times with an average gold dissipation of 16.4 % into larger sieve fractions.



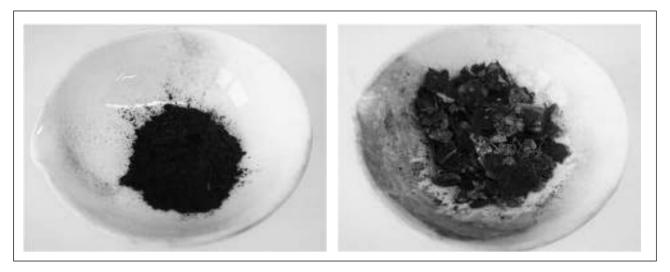


Figure 5: Sieve fraction  $< 90 \mu m$  (left) and  $> 90 \mu m$  (right) from pyrolized computer PCB's

TS leaching experiments were conducted on comminuted (< 500 µm) and pyrolized specimens (<90 μm). The best leaching experiments showed a dissolution of just 45.8 % gold 32.9 % silver and 46.2 % copper (0.2 M TS, 0.4 M NH<sub>3</sub>, 20 mM Cu, s/l 1/20, 40 °C, 24 – 72 h) with pyrolized material, while comminuted e-waste samples showed a significantly lower gold recovery. The reason for insufficient precious metal recovery lies in the reagent consumption caused by base metal dissolution. Considering the partially dissolved metals Cu, Ni and Zn from pyrolized PCBS's, the base metal dissolution consumes 0.32 - 0.40 mol thiosulfate and ammonia. Taking into account that also oxidative degradation of TS takes place, the available reagents for gold dissolution are strictly limited. Unlike the pyrolized samples, copper and other ductile metals were not separated by sieving. The higher concentration of metallic impurities causes an increased reagent consumption, when compared with pyrolized materials. The 48 and 72 h leaching experiments revealed a decreasing recovery of precious metals with time. This effect is most likely due to precipitation caused by electrochemical reduction through less noble metals or may lie in the degradation of thiosulfate with time in the oxidative environment. Pyrolized PCBs shows another significant drawback for the TS leaching of gold: very fine and reactive carbon matter causes a precipitation of gold. Different researchers used this mechanism for gold recovery from leaching solutions [39, 40]. The gold loss due to adsorption on carbon and precipitation was proved by addition of fine carbon powder into a gold containing TS solution. As a result, the filtered solid residue showed a significant gold content.

To reduce or avoid a reagent consumption and degradation, comminuted and pyrolized e-waste specimens were leached with different mineral acid solutions to separate base metals. The experiments showed, that a complete base metal separation is not possible within one leaching step, even though hydrogen peroxide is added into the aggressive leaching solutions (Figure 6). The variety of metals react differently depending on the applied leaching system. At the same time the high metal concentration binds complexing agents. Experiments aiming on the investigation of leaching time revealed the decreased yield of noble metals, in particular Cu and Sn, while less noble metals remain constant or increase. This effect was also observed for gold in other leaching system. However, the base metal



extraction did not improve the gold dissolution in subsequent TS leaching significantly, but the silver recovery was increased after hydrochloric leaching (47.9 % compared to 32.9 % without base metal separation). The reason for the increased recovery lies in the dissolution of Sn-based solder alloys and thus the liberation of silver.

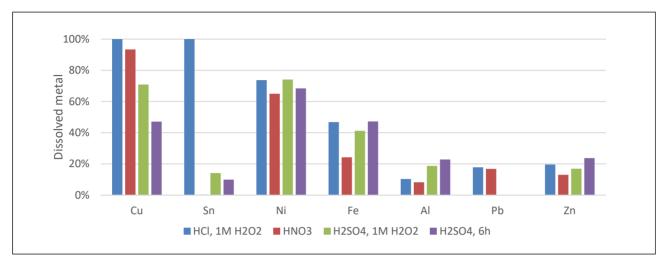


Figure 6: Selected results of base metal leaching of pyrolized PCB's (2 M reagent concentration and 1 M  $H_2O_2$  each, 85 °C, s/l = 1/20, rpm 300)

## Direct Thiosulfate Leaching of Untreated E-Waste Specimens

Many experiments with superficial TS leaching of untreated and non-comminuted e-waste samples showed quite deceiving results. As can be seen in Figure 7, no gold seems to be present on the surface after leaching anymore. But the chemical analysis of the leaching solution reveals that gold was not dissolved. The surface was examined and treated carefully with diluted acidic solutions, resulting in the appearance of gold below the dark matter. A surface analysis via EDS revealed that the dark layer consists of copper, gold and sulfur, which fits with the color Cu sulfide or sulfate compounds [41, 42]. The formation of sulfides takes place due to oxidative degeneration of thiosulfates to polythionates or sulfates. These compounds can form insoluble complexes, which precipitate on the gold surface and create a stable passive layer hindering further gold dissolution. This effect is facilitated by high copper or oxygen concentrations over a longer period and a fluctuating pH value [43, 44].



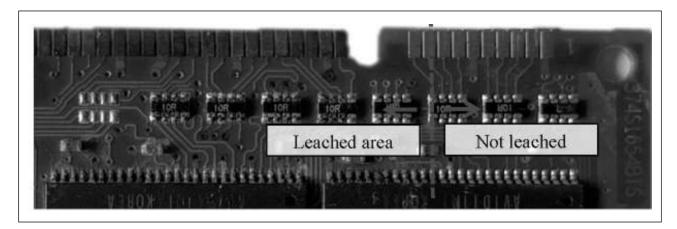


Figure 7: Partially leached RAM (0.2 M TS, 0.3 M NH<sub>3</sub>, 0.05 M CuSO<sub>4</sub>, 25 °C, 24 h)

On the basis of first experiments resulting in a surface passivation, the leaching conditions adapted and strictly controlled. To prevent the effect of oxidative TS degradation following measures took place: Reduction of copper addition to  $0.02\,\mathrm{M}$ ; stabilization of Cu compounds via increased ammonia addition of up to  $0.6\,\mathrm{M}$  (TS: NH $_3$  = 1:3-1:6); use of a closed leaching reactors to lower the oxygen concentration and ammonia evaporation. As a result the leaching efficiency could be increased significantly. Within the first 6 h less than 20 % gold was recovered, while after 24 h more than 80 % and after 48 h up to 98.9 % gold were dissolved at  $0.1-0.2\,\mathrm{M}$  TS and  $21-30\,^{\circ}\mathrm{C}$ . At this parameter set it was found, that time has a significant influence on gold recovery but also on the selectivity (Figure 8). The lower copper and oxygen concentration bring a weaker oxidative environment for gold dissolution. Between 24 and 48 gold is dissolved nearly completely. Since the subjacent metals appear to the surface, their co-dissolution is facilitated. Soldering alloys were not dissolved by TS, so that silver was bound in that phase and could not be dissolved. Hence just an average of 7.64 % of contained silver could be extracted in leaching experiments without mechanical-physical or other pretreatment methods.

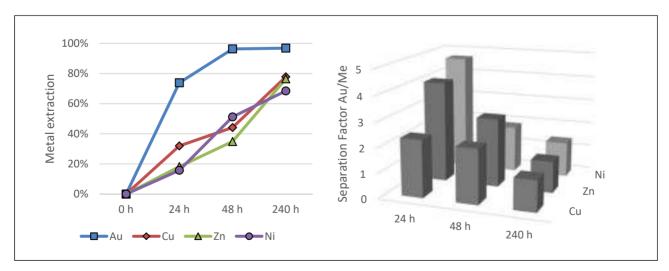


Figure 8: Metal extraction (left) and selectivity of selected TS leaching experiments (0.1 - 0.2 M) TS,  $0.6 \text{ M NH}_3$ ,  $0.02 \text{ M CuSO}_4$ ,  $25 ^{\circ}\text{C}$ 



## Conclusion

Leaching experiments with flue dust from e-waste shredders proved a low selectivity for thiosulfate in multi-metal systems. In an experimental series of more than 40 trials, the average gold yield was 15 % or lower, while also copper, silver and other base metals were dissolved to a certain extent. The best results could be achieved via air and oxygen injection into the leaching slurry. But these aeration trials just enabled a recovery of up to 23.3% Au, 39.9 % Ag and 61.8 % Cu were dissolved (0.1 M TS, 0.4 M NH<sub>3</sub>, 10 mM Cu, 25 °C, s/l = 1/20 t = 6 h). Metals like Cu, Zn and Ni but also other base metals cause a significant reagent consumption (> 0.12 mol in a 0.1 M TS solution).

Mechanical-physical and thermal pre-treatment of e-waste may have some advantages for the gold recovery in general, for instance through liberation of covered gold components and separation of other materials but come along with a severe drawback: the higher presence of base metals and the higher reagent consumption which impede the gold recovery. An aforegoing base metal separation could enhance the leaching conditions but requires a multiple-step leaching procedure with aggressive solutions. The best leaching experiments with pre-treated e-waste samples showed a recovery of 45.8 % gold 32.9 % silver and 46.2 % copper (0.2 M TS, 0.4 M NH<sub>3</sub>, 20 mM Cu, 40 °C, s/l = 1/20, t = 24 h) with pyrolized material.

Superficial leaching of holistic electronic components like RAM's has the highest potential, although a strict parameter control has to take place. Untreated electronic components just show a very limited amount of metal on the surface, from which only the metals used for electrical contacts have a notable solubility in thiosulfate solutions. Inadequate process conditions cause a precipitation of thiosulfate compounds and a gold passivation. With a gold recovery of > 85 %, the best results for superficial leaching trials were achieved at 0.1 - 0.2 M thiosulfate, 0.2 - 0.6 NH<sub>3</sub>, 0.02 M Cu at 21 - 30 °C within 24 - 48 h. It was found that the effect of copper concentration is strongly depending on leaching duration but also on thiosulfate and ammonia concentration. A high Cu content accelerates the gold dissolution but also enhances the oxidative thiosulfate degradation, which could cause the precipitation of gold. This correlation needs further investigation, to enable an efficient gold recovery at a reduced leaching duration.

In conclusion, hydrometallurgical gold recovery from electronic waste by cyanide alternative leaching reagents still needs further research. Thiosulfate is the only alternative reagent developed into commercial application for primary gold production, but literature on gold recovery form electronic waste is strongly limited. The conducted experiments revealed, that thiosulfate leaching can enable an efficient gold recovery but shows many drawbacks in terms of reagent stability and sensibility for metal interactions. The reaction mechanism and interdependence of thiosulfate with elements contained in electronic waste needs further investigation.



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