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Study of the influence of temperature and duration of chlorinating roasting on the extraction of gold from E-waste

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ABSTRACT

In this work, based on experimental studies, the effect of roasting temperature and duration on the chlorination of gold from E-waste under low-temperature conditions was studied. It has been established that in the low-temperature range of 150-300 °C, the gold content in the residue shows a sharp decrease, reaching its minimum of 8 ppm at 250 °C. A further increase in temperature does not affect the gold content in the cinder. It has been shown that during chlorinating roasting above a temperature of 250 °C, side reactions should be expected to occur with the formation of additional phases representing chlorides of copper, iron and other elements. The results of SEM and XRD analyzes of the solid phase obtained after chlorinating roasting at a temperature of 250 °C and a roasting duration of 20 minutes showed the presence of copper and iron chlorides in them. Optimal technological parameters and modes of the process of chlorinating roasting of E-waste with gaseous chlorine have been established: temperature – 250 °C; roasting duration – 20 minutes; chlorine consumption is 1.5 times more than the stoichiometrically required amount (SRA) for gold chlorination. Under optimal roasting conditions, a high, up to ~97.6%, recovery of gold in the form of gold chloride ($AuCl_3$) was achieved. The results of the experiments can be a basic basis and make it possible to conduct more representative experiments (scaling) in the future, taking into account the established optimal technological parameters. This will provide a chance to evaluate the possibility of extracting gold from E-waste of different types and compositions and to conduct further research on obtaining pure gold from the collected representative amount of gold sublimate $AuCl_3$ by smelting.

Keywords: E-waste, gold, roasting, $AuCl_3$ sublimate, temperature, recovery.

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Introduction

Increasing environmental requirements necessitate the introduction of new accessible technologies with the transition to a circular economy. The issues of resource-saving of primary raw materials and reducing the costs of metal production are the main tasks nowadays, the solution of which requires finding new types of sources of raw materials for the production of metals. One of the promising areas is the involvement of secondary raw materials in the processing, which is becoming widespread in European countries where there is a clear shortage of high-quality primary raw materials. In recent years, the recycling of waste electronic and electrical equipment, combined into a separate category, and referred to in the scientific literature as electronic waste (E-waste) [[1], [2], [3], [4], [5], [6], [7]], has received high interest and development.

Known technologies for processing E-waste, including pyro-, hydro- and biometallurgical technologies, require serious modernization, both in terms of technology and environmental requirements [[8], [9], [10], [11], [12], [13]]. The lack of rational technology for processing E-waste has led to its growth in terms of volume. According to experts [[14], [15], [16]], up to 343 thousand tons of electronic waste are accumulated in Kazakhstan per year, which could be used as an additional source of raw materials for the production of valuable metals.

The use of chlorinating technologies for processing E-waste, which is currently widely used for the extraction of valuable metals from various types of primary raw materials and technogenic waste [[17], [18], [19], [20]], has a high theoretical and practical interest. The results of systematic studies carried out in [19] showed the possibility of selective extraction of gold from various types and compositions of primary gold-bearing raw materials at a relatively low temperature. The positive results obtained by the authors indicate the prospects of using chlorine gas as a chlorinating reagent for the sublimation of gold in the form of its sublimate ($AuCl_3$) from E-waste. However, for a more detailed assessment, data on the completeness and rate of interaction of gold with chlorine is important. Knowledge of these factors is necessary to study the mechanism of the process of chlorination of E-waste with chlorine gas.

This research [20] on the interaction of E-waste components with gaseous chlorine shows the fundamental thermodynamic possibility of selective gold extraction from E-waste. Significant factors affecting gold recovery during low-temperature chlorinating roasting of E-waste with chlorine gas are chlorine consumption, temperature, process duration and impurity content. At low temperatures (100-250 ^{0}C), only $AuCl_3$ has high vapor pressure. Therefore, it can be argued that the main factors influencing the completeness of gold extraction from E-waste will be the temperature and duration of the process. In this case, the consumption of chlorine should be optimal, and it should be spent on the chlorination of gold, the content of which, as the literature data show, in E-waste can vary from 50 to 350 ppm, depending on the type and composition of electronic scrap.

To obtain quantitative data on the recovery and rate of gold chlorination, this article contains studies on the chlorination of gold from E-waste in a stream of chlorine gas. The effect of roasting temperature and time on the chlorination of gold and its extraction from E-waste in the form of a volatile sublimate $AuCl_3$ was studied.

Materials and research methods

Computer boards were used as the initial object. -35 mesh PC board materials were prepared in advance using an SM 300 cutting mill from Retsch GmbH. The gold content in the source material is 310 ppm.

Roasting was carried out in a temperature-controlled laboratory electric furnace Nabertherm HTC-03/15/B170 at a temperature of 250 ^{0}C in an atmosphere of excess chlorine gas. The heating rate of the furnace was constant and amounted to 12 ^{0}C/min.

In all experiments, the gas flow (chlorine), to optimize it, was constant and amounted to 150 ml/min. The consumption of chlorine was 1.5 times higher than the stoichiometrically required amount (SRA) for the chlorination of gold according to the reaction: $2Au + 3Cl_2 = 2AuCl_3$.

Experiments were carried out at temperatures of 150, 200, 250 and 300 ^{0}C. The experimental time varied from 10 to 20 minutes in 5-minute increments. The experimental methodology is based on the experimental planning theory method, which makes it possible to determine the influence of temperature and process duration on the extraction of gold from E-waste under conditions of chlorination with chlorine.

The total number of experiments was 16 experiments. Each experiment was repeated three times for reproducibility to obtain accurate results. At the end of each experiment, the weight of the residue was monitored. The presence of gold in the residues obtained after roasting was determined based on the results of elemental, SEM and XRD analyses. The gold content in the residue was additionally determined by the traditional method of chemical analysis.

Based on the results of experimental data, the values of gold recovery from E-waste at different temperatures and roasting times were calculated.

Diagram of the laboratory setup and experimental methodology

The general diagram of the laboratory installation for gold extraction is shown in Fig. 1.

The experimental procedure was as follows. The initial sample was loaded into boat crucible 4 and placed in a quartz reactor 2 installed in the zone of a uniform temperature field of electric furnace 1. The working area of the furnace allows for placing four boat crucibles at the same time, which can increase the amount of raw material being roasted. In our case, we used one boat crucible with a minimum weight of 15 g.

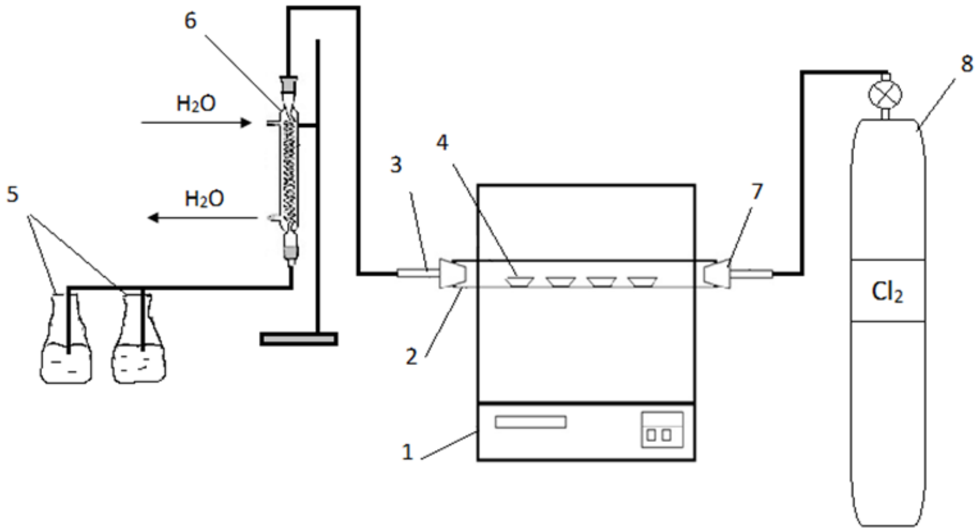


Figure 1 – General diagram of the laboratory installation to extract gold from E-waste:
1 – Nabertherm HTC-03/15/B170 electric furnace; 2 – quartz reactor;
3 – gases containing AuCl3 sublimate; 4 – boats crucible for samples;
5 – exhaust gas absorbers; 6 – AuCl3 sublimate trapping device;
7 – plug with a hole for supplying chlorine to the reactor;
8 – cylinder with chlorine (gas). *The picture was illustrated by the authors.*

The quartz reactor is plugged on opposite sides with a plug, which has holes for the input and output of gases. The roasting was carried out in a stream of chlorine gas, which was supplied from cylinder 8 through a plug with a hole 7 into a quartz reactor 2.

Sublimate ($AuCl_3$) formed during roasting was removed from the reactor through pipeline 3 and captured in a specially designed device 6. Continuous removal of the sublimate from the reaction zone ensured that the gold chlorination reaction proceeded practically in the absence of its chlorides in the reaction zone.

Exhaust gases containing chlorine were captured in absorption flasks containing lime milk. At the end of the experiment, the furnace was turned off. The solid residue obtained after the experiment was removed from the furnace, weighed and subjected to chemical and phase analysis. The degree of gold chlorination under various experimental conditions was determined by the difference, taking into account the amount of the initial sample and the resulting residue and the gold content in them.

Results and discussion

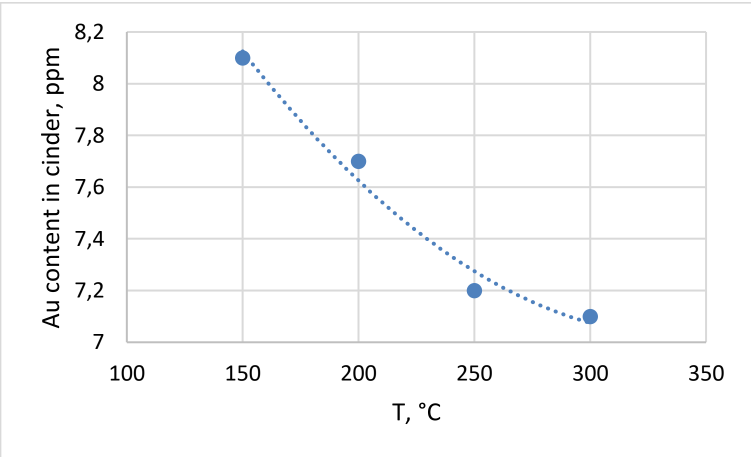
The results of experiments on low-temperature chlorination of E-waste with gaseous chlorine are given in Table 1.

Figure 2 shows the dynamics of changes in the gold content in the source material depending on the temperature and duration of roasting.

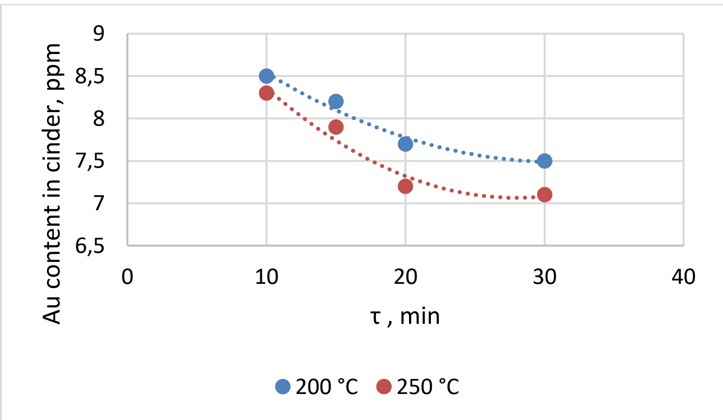
It is easy to notice that in the low-temperature range of 150-300 °C, the nature of the curve of the dependence of the gold content in the residue on temperature shows a sharp decrease in the gold content, reaching its minimum of 8 ppm at 250 °C (Fig. 2 (A)). A further increase in temperature does not affect the gold content in the cinder. This indicates that during chlorinating roasting above a temperature of 250 °C, side reactions should be expected to occur with the formation of additional phases representing chlorides of copper, iron and other elements. In particular, as shown by the results of SEM and XRD analyses, copper and iron chlorides were found in the residues obtained after chlorinating roasting at a temperature of 250 °C and a roasting time of 20 minutes. The obtained results are in good agreement with the results of thermodynamic analysis of the reactions of interaction of E-waste components with gaseous chlorine, where during chlorinating roasting of E-waste at low temperatures (below 400 °C), a high thermodynamic probability of interaction of non-ferrous metals (Cu, Pb, Zn) was established with chlorine gas to form solid chlorides.

Table 1 - Results of experiments on chlorination of E-waste with chlorine

#	Initial weight, g.	Temperature, ºC	Time, min	Quantity of residue, g.	Content of Au in the residue, ppm
1	200	150	10	73	8.8
2	200	150	15	65	8.4
3	200	150	20	54	8.1
4	200	150	30	49	7.9
5	200	200	10	71	8.5
6	200	200	15	67	8.2
7	200	200	20	57	7.7
8	200	200	30	50	7.5
9	200	250	10	70	8.3
10	200	250	15	64	7.9
11	200	250	20	99	7.2
12	200	250	30	52	7.1
13	200	300	10	68	8.4
14	200	300	15	54	7.8
15	200	300	20	49	7.1
16	200	300	thirty	46	7



A)



B)

Figure 2 – Dependence of gold content in the residue on temperature (A) at τ = 20 min., and roasting duration (B)

From Fig. 2 (B) it is clear that the duration of roasting is an equally important parameter affecting the chlorination of gold: an increase in the roasting time also leads to a decrease in the gold content in the residue obtained after roasting. At T = 200 °C and roasting time τ = 20 minutes, the gold content in the residue is reduced from 310 ppm to 9.5 ppm. The greatest effect of reducing gold in the residue is achieved at T = 250 ^{0}C and roasting time τ = 20 minutes: the gold content in the residue is 8 ppm.

The most significant technological indicator of the chlorination roasting process is the extraction of gold from E-waste under experimental conditions. Based on the task set, using the results of experiments, the values of gold extraction into sublimates in the form of AuCl3 sublimate were calculated.

Gold recovery was calculated using the formula:

$$\alpha = 100 - \left(\frac{Me_{cinder}}{Me_{scrap}} * 100\right),$$

where: Me $_{cinder}$ – metal content in the cinder, %;

Me $_{scrap}$ – metal content in the original scrap, %.

Figure 3 shows the graphical dependences of gold extraction (α) on temperature and roasting duration, developed from the results of experimental data.

The recovery of gold from E-waste increases with increasing temperature. Moreover, its sharp increase, at a constant roasting time, is observed in the temperature range of 100-250 ^{0}C, reaching its maximum at 235 ^{0}C (τ = 20 min.) equal to ~97.6% (Fig. 3 (A)). A further increase in temperature does not affect gold extraction; on the contrary, a slight decrease is observed, which is associated with a change in the phase composition of the starting material during the roasting process. At high roasting temperatures (more than 250 ^{0}C), sintering of the material occurs with the formation of additional phases: $CuCl_2$; FeClO and $Cu_2Cl(OH)_3$, which inhibit the interaction of gold with chlorine and reduce the reaction rate. However, as experimental results show, the greatest reduction of gold with chlorine is achieved up to a temperature of 250 ^{0}C. Further chlorination of gold can be achieved by increasing the temperature, however, the resulting gold sublimate will decompose into metallic gold and chlorine to the point of capture. In addition, at elevated temperatures, we should expect changes in the composition of the gas phase due to the volatilization of undesirable accompanying metals (As, Cd, etc.), as well as organics due to the thermal destruction of plastics, etc.

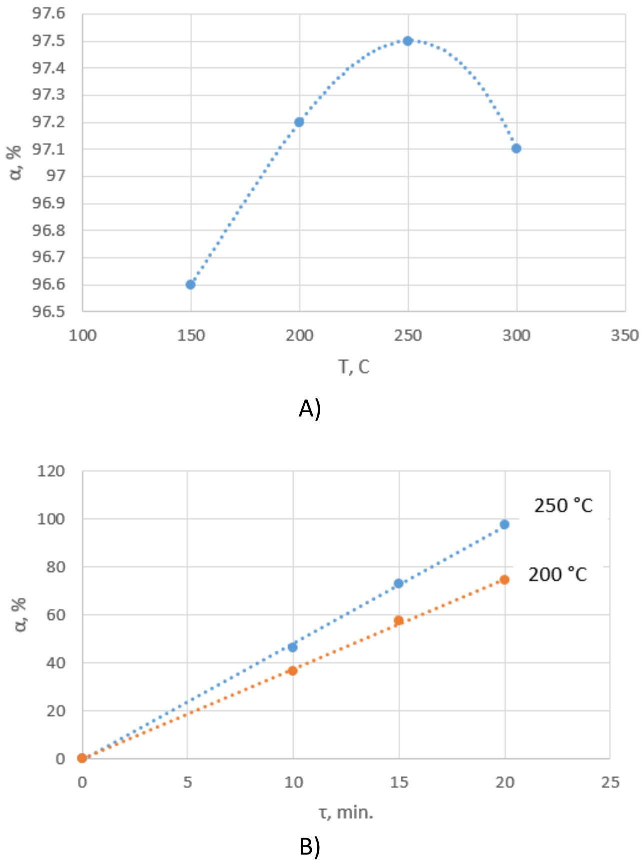


Figure 3 – Dependence of gold recovery (α) on temperature (A) at τ = 20 min., and duration (B)

Increasing the experiment time, at a constant temperature, has a significant effect on gold recovery in the time range from 10 to 20 minutes, sharply increasing gold recovery during roasting to a maximum of 20 minutes (Fig. 3 (B)). Increasing the temperature reduces the roasting time and increases the gold recovery, indicating that the roasting time has a negligible effect on gold recovery from E-waste.

The obtained positive experimental results provide a chance to select the optimal technological parameters and modes of the process of chlorinating roasting of E-waste with gaseous chlorine: temperature – 250 ^{0}C; roasting duration – 20 minutes; chlorine consumption is 1.5 times higher than its stoichiometric required amount for chlorination of gold. The established parameters provide high gold recovery up to ~97.5% from E-waste in the form of $AuCl_3$ sublimate.

The results of the experiments can serve as a basic basis to conduct more representative experiments (scaling) in the future, taking into account the established optimal technological parameters. This will allow us to evaluate the possibility of extracting gold from E-waste of different types and compositions and to conduct further research on obtaining pure gold from the collected representative amount of gold sublimated $AuCl_3$ by smelting.

Conclusions

Based on experimental studies, the influence of roasting temperature and time on the chlorination of gold from E-waste has been established. Extraction from E-waste, at a constant roasting time, increases with increasing temperature and reaches its maximum equal to ~97.6% at 235 ^{0}C (τ = 20 min.). A further increase in temperature does not affect gold extraction; on the contrary, a slight decrease is observed.

At a constant temperature, increasing the duration of the experiment from 10 to 20 minutes linearly increases the extraction of gold during roasting. Increasing the temperature reduces the roasting time and shifts gold recovery towards higher levels.

Optimal technological parameters and modes of the process of chlorinating roasting of E-waste with gaseous chlorine have been established: temperature – 250 ^{0}C; roasting duration – 20 minutes; chlorine consumption is 1.5 times more than SRA for gold chlorination. Under optimal roasting conditions, a high, up to ~97.6%, recovery of gold in the form of gold chloride ($AuCl_3$) was achieved.

The obtained data will be used for further research on the extraction of gold from E-waste of various types and compositions in the form of $AuCl_3$ sublimate and the isolation of pure gold from it.

Conflict of interest. On behalf of all authors, the corresponding author confirms that there is no conflict of interest.

CRediT author statemen: **E.Zoldasbay:** Supervision, Conceptualization, Methodology. **A. Argyn:** Data curation, Writing- Original draft preparation. **M.Kurmanseitov:** Investigation. **K.Dosmukhamedova:** Investigation, Software, Validation: **G.Daruesh:** Writing- Reviewing.

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Хлорлап күйдіру кезінде Е-қалдықтарынан алтынды бөліп алуға температура мен процес ұзақтығының әсерін зерттеу

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ТҮЙІНДЕМЕ

Бұл жұмыста эксперименталды зерттеулер негізінде төмен температура жағдайында күйдіру температурасы мен уақытының Е-қалдықтардан алтынның хлорлануына әсері зерттелді. 150-300 °С аралығындағы төмен температурада қалдықтағы алтынның мөлшері 250 °С температурада 8 ppm-ге тең минимумға жете отырып, күрт төмендейтіні анықталды. Температураның одан әрі өсуі өртенді құрамындағы алтынға әсер етпейді. 250 °С-тан жоғары температурада хлорлап күйдіру кезінде жанама реакциялардың жүруі нәтижесінде қосымша фазалар мыс, темір және басқа элементтердің хлоридтерінің түзілетіні анықталды. 250 °С температурада және 20 минут хлорлап күйдіру уақытынан кейін алынған қатты фазаны SEM және XRD талдауларының нәтижелері оларда мыс және темір хлоридтерінің барын көрсетті. Е-қалдықтарды хлор газымен хлорлау процесінің оңтайлы технологиялық параметрлері мен режимдері белгіленді: температура - 250 °C; күйдіру ұзақтығы – 20 мин.; хлор шығыны алтынды хлорлау үшін СҚМ-нен 1,5 есе көп. Оңтайлы күйдіру жағдайында жоғары ~97,6% дейін алтын хлорид ($AuCl_3$) түрінде бөліп алынды. Жүргізілген тәжірибелердің нәтижелері базалық негіз бола алады және белгіленген оңтайлы технологиялық параметрлерді ескере отырып, одан әрі өкілетті эксперименттерді (масштабтау) жүргізуге мүмкіндік береді. Бұл әртүрлі типтегі және құрамдағы Е-қалдықтардан алтынды бөліп алу мүмкіндігін бағалауға және жиналған алтын сублиматының $AuCl_3$ өкілетті мөлшерінен балқыту арқылы таза алтын алу бойынша одан әрі зерттеулер жүргізуге мүмкіндік береді.

Түйін сөздер: Е-қалдықтар, алтын, күйдіру, $AuCl_3$ сублиматы, температура, бөліп алу.

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Исследование влияния температуры и продолжительности хлорирующего обжига на извлечение золота из Е-отходов

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АННОТАЦИЯ

В работе на основании экспериментальных исследований изучено влияние температуры и времени обжига на хлорирование золота из Е-отходов в условиях низких температур. Установлено, что в интервале низких температур 150-300 °C содержание золота в остатке показывает резкое снижение с достижением своего минимума, равного 8 ppm, при 250 °C. Дальнейший рост температуры на содержание золота в огарке не влияет. Показано, что в процессе хлорирующего обжига выше температуры 250 °C следует ожидать протекания побочных реакций с образованием дополнительных фаз, представляющих хлориды меди, железа и других элементов. Результаты SEM и XRD-анализов твердой фазы, полученной после хлорирующего обжига при температуре 250 °C и времени обжига 20 минут, показали наличие в них хлоридов меди и железа. Установлены оптимальные технологические параметры и режимы процесса хлорирующего обжига Е-отходов газообразным хлором: температура – 250 °C; продолжительность обжига – 20 мин.; расход хлора в 1,5 раза больше СНК для хлорирования золота. В оптимальных условиях ведения обжига достигнуто высокое, до ~97,6 %, извлечение золота в виде ее хлорида ($AuCl_3$). Результаты проведенных опытов могут служить базовой основой и позволяют в дальнейшем провести более представительные эксперименты (масштабирование) с учетом установленных оптимальных технологических параметров. Это позволит оценить возможность извлечения золота из различного по типу и составу Е-отходов, и провести дальнейшие исследования по получению чистого золота из собранного представительного количества сублимата золота $AuCl_3$ путем плавки.

Ключевые слова: Е-отходы, золото, обжиг, сублимат $AuCl_3$, температура, извлечение.

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