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Effect of ultrasonic treatment on tin recovery from decommissioned displays in sulphuric, hydrochloric, and methanesulphonic acid solutions

E.B. Kolmachikhina, O.B. Kolmachikhina, Ya.A. Yankina, Z.M. Golibzoda, P.A. Brizhevataya, D.S. Sedel'nikova, R.E. Khabibulina

Ural Federal University n.a. the First President of Russia B.N. Eltsin
19 Mira Str., Ekaterinburg 620002, Russia

✉ Elvira B. Kolmachikhina (e.b.khazieva@urfu.ru)

Abstract: The study investigates the physicochemical patterns of tin leaching from the surface of glass substrates from decommissioned displays in hydrochloric, sulphuric, and methanesulphonic acids. The effects of acid concentration (0.1–1.0 N), duration (10–60 min), temperature (298–353 K), and ultrasonic treatment intensity (UST) (120–300 W/cm^2) on leaching performance were evaluated. It was demonstrated that ultrasonic treatment positively impacts sulphuric acid leaching of tin, increasing its recovery by 14–16 %. However, during leaching in hydrochloric and methanesulphonic acid solutions, UST led to a reduction in tin recovery to 28 % and 1.7 %, respectively, due to acid decomposition under ultrasound. The partial reaction orders for tin leaching in HCl, H_2SO_4, and CH_3SO_3H were determined to be 0.8, 1.4, and 1.1, respectively, and changed to 1.5, 1.1, and 0.3 under ultrasound for the corresponding acids. An increase in temperature from 298 K to 333 K significantly improved tin recovery in sulphuric and hydrochloric acids. However, raising the temperature to 353 K led to a decrease in tin ion concentration after 10–20 min, likely due to tin hydrolysis and precipitation. The calculated apparent activation energies of tin oxide dissolution in HCl solutions were 40.4 kJ/mol without UST and 22.9 kJ/mol with UST. For H_2SO_4, the apparent activation energy was 4.0 kJ/mol, increasing to 29.0 kJ/mol under ultrasonic treatment. Therefore, the study showed that tin leaching from glass substrates of decommissioned displays proceeds in a kinetic regime when HCl is used and in a diffusion regime in H_2SO_4 solutions, with ultrasonic treatment facilitating the transition to a mixed regime.

Keywords: tin, sulphuric acid, hydrochloric acid, methanesulphonic acid, leaching, ultrasound.

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Влияние ультразвуковой обработки на выщелачивание олова из отслуживших дисплеев в растворах серной, соляной и метансульфоновой кислот

Э.Б. Колмачихина, О.Б. Колмачихина, Я.А. Янкина, З.М. Голибзода, П.А. Брижеватая, Д.С. Седельникова, Р.Э. Хабибулина

Уральский федеральный университет (УрФУ) им. первого Президента России Б.Н. Ельцина
Россия, 620002, г. Екатеринбург, ул. Мира, 19

✉ Эльвира Барыевна Колмачихина (e.b.khazieva@urfu.ru)

Аннотация: Изучены физико-химические закономерности выщелачивания олова с поверхности стеклянных подложек отслуживших дисплеев в соляной, серной и метансульфоновой кислотах. Установлено влияние концентрации указанных кислот (0,1–

1,0 н), продолжительности (10–60 мин), температуры (298–353 K) и интенсивности ультразвуковой обработки (УЗО) (120–300 Вт/см^2) на показатели выщелачивания. Показано, что ультразвуковое воздействие оказывает положительное влияние на сернокислотное выщелачивание олова, позволяя повышать его извлечение на 14–16 %. При выщелачивании в растворах соляной и метансульфоновой кислот УЗО приводила к снижению извлечения олова до 28 и 1,7 % соответственно, что связано с их разложением под действием ультразвука. Частные порядки реакций выщелачивания олова по HCl, H_2SO_4 и CH_3SO_3H составили 0,8, 1,4 и 1,1, при воздействии ультразвука частные порядки изменялись следующим образом для соответствующих кислот: 1,5, 1,1 и 0,3. Увеличение температуры с 298 до 333 K значительно повышало извлечение олова в серной и соляной кислотах. Повышение температуры до 353 K приводило к снижению концентрации ионов олова через 10–20 мин процесса, что, вероятнее всего, связано с гидролизом и осаждением олова. Рассчитанные величины кажущейся энергии активации растворения оксида олова без и с использованием УЗО в растворах HCl составили 40,4 и 22,9 кДж/моль соответственно. В случае использования H_2SO_4 кажущаяся энергия активации составила 4,0 кДж/моль, а при акустическом воздействии – 29,0 кДж/моль. Таким образом, проведенные исследования показали, что выщелачивание олова из стекол отслуживших дисплеев протекает в кинетическом режиме при использовании HCl и в диффузионном режиме в растворах H_2SO_4, а УЗО способствует переходу процессов в смешанный режим.

Ключевые слова: олово, серная кислота, соляная кислота, метансульфоновая кислота, выщелачивание, ультразвук.

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Introduction

One of the main types of decommissioned electronic devices consists of screens and monitors. This category accounts for approximately 10 % of the total mass of generated electronic waste. Nearly all screens and monitors contain indium tin oxide (ITO) in their displays. ITO (90 % In_2O_3, 10 % SnO_2) is transparent to visible light, possesses electrical conductivity, and is used as a conductive layer applied to the glass substrate of the display [1]. Additionally, ITO is widely used in the production of solar panels, optical coatings, antistatic films, and thermal protection.

Approximately 75 % of the ITO produced is used in the manufacture of flat panel displays. These displays consist of multiple layers, including polarisation filters, glass substrates, and liquid crystals. A layer of ITO, 50—200 nm thick, is applied to the surface of the glass substrate [2], while a polariser made of polyvinyl acetate film is attached to the opposite side. The indium content in the glass substrates ranges from 100—350 mg/kg [3, 4], and the tin content ranges from 25—60 mg/kg [5, 6]. Although the indium content in glass substrates is relatively low, it is comparable to that found in zinc concentrates extracted from sulphide ores [7]. This makes decommissioned monitors a highly promising secondary source of indium.

Tin is a scarce metal [8, 9], classified as a strategic metal in many countries. With the accelerating development of the semiconductor and electric vehicle industries, tin consumption has also increased. Recovering tin from secondary sources will help alleviate the shortage of mineral resources and reduce environmental pollution.

In studies focused on the recycling of liquid crystal displays, the emphasis is often placed on the behaviour of indium due to its high content in ITO. Although the tin content in ITO is relatively low, exploring ways to extract it will provide a comprehensive solution for recycling decommissioned displays.

The recycling of glass substrates begins with pre-treatment, which may include steps such as polariser removal, crushing, grinding, and beneficiation [10—13]. Subsequent metallurgical processing can be carried out using pyrometallurgical methods (reduction and distillation of indium and tin compounds) [14] and/or hydrometallurgical methods (leaching, liquid recovery, cementation, etc.) [15—19].

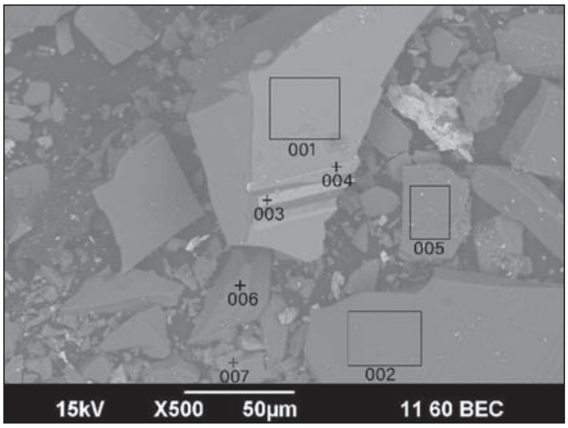
In this study, a comparative assessment of the tin leaching rate from the surface of glass substrates in hydrochloric, sulphuric, and methanesulphonic acids was conducted. The acids used in the study are strong acids and can be arranged in the following order based on increasing strength, as expressed by the dissociation constant (pK_{d1}): CH_3SO_3H (–1.86), H_2SO_4 (–3), HCl (–7) [20—22]. Ultrasonic treatment (UST) was used to accelerate the ITO leaching process, providing thermal, mechanical, and sonochemical effects [23—30].

The aim of this work was to investigate the influence of ultrasonic treatment, temperature, concentration, and type of acids on the physicochemical patterns of tin dissolution from the surface of crushed glass substrates from decommissioned liquid crystal displays.

Experimental methodology

Researmh materials, equipment, and methods

The glass substrates from decommissioned liquid crystal displays were pre-washed with water, air-dried, and then heated to 463 K to soften and manually remove the film. The cleaned glass substrates were crushed in a rod mill. A fraction of the crushed material smaller than 1 mm was separated by sieving and used for further experiments. After thoroughly homogenising the material, a representative sample was taken by quartering for subsequent chemical analysis using an atomic absorption spectrophotometer (AAS) "novAA 300" (Analytik Jena, Germany). The particle size distribution of the material was determined using a HELOS&RODOS laser diffraction particle size analyser (Sympatec GmbH, Germany). The morphology of the crushed material samples and their chemical composition were examined with a JSM-6390LA scanning electron microscope (JEOL, Japan), equipped with a JED-2300 energy-dispersive microanalysis system (JEOL, Japan).



Element	Measurement area number						
	001	002	003	004	005	006	007
Mg	2.1	1.7	0.9		1.6		4.0
Al	10.3	19.0	5.0	23.3	18.3	12.8	19.8
Si	77.8	67.2	73.2	61.7	67.8	48.8	69.5
Ca	2.9	12.1	2.1	1.3	12.3	38.5	6.7
Cr	–	–	12.7	–	–	–	–
Fe	–	–	3.2	–	–	–	–
Mo	–	–	2.3	13.7	–	–	–
In	5.5	–	–	–	–	–	–
Sn	1.3	–	–	–	–	–	–

Fig. 1. SEM image and elemental composition (%) of crushed glass substrates from displays

Рис. 1. СЭМ-изображение и элементный состав (%) измельченных стеклянных подложек дисплеев

The majority of the material (>85 %) consisted of irregularly shaped particles smaller than 300 μm (Fig. 1). Elemental analysis, along with previous studies, showed that the material was primarily composed of aluminium, silicon, and calcium oxides. Indium and tin compounds were detected on the glass surface (Fig. 1, point *1*), while molybdenum compounds were found on the surface of the conductive tracks (Fig. 1, points *3* and *4*). The crushed glass substrates from the displays contained the following concentrations, in mg/kg: indium — 174.8, tin — 1.7.

The experiments used sulphuric acid (H_2SO_4, chemically pure), hydrochloric acid (HCl, chemically pure), and methanesulphonic acid (CH_3SO_3H, chemically pure). The initial acid solutions were prepared by diluting concentrated acids with stirring on a magnetic stirrer.

Ultrasonic treatment was performed using the UZTA-0.1/28-O apparatus (LLC Ultrasound Technology Centre, AltSTU, Biysk), equipped with an immersion-type emitter, at a frequency of 28 ± 2.5 kHz and ultrasonic intensity (I) ranging from 120 to 300 W/cm^2.

Crushed glass leaching

Leaching experiments were conducted in a thermostatically controlled reactor with a volume of 0.5 dm^3 at a temperature of $T = 298{\div}353$ K, a duration of $\tau = 10{\div}60$ min, and acid concentrations of 0.1—1.0 N. The liquid-to-solid ratio (L : S) was 10 : 1 (cm^3/g).

Acid solutions with the required concentration were poured into the reactor, sealed, and heated to the desired temperature under constant stirring. Then, the ultrasonic emitter was immersed in the reactor to a fixed depth, 20 g of material was loaded, and the ultrasonic generator was turned on, with the pulp temperature being automatically maintained with an accuracy of ±2 K.

After leaching, the pulp was filtered, and the cake was washed with distilled water. The cake was then subjected to acid treatment at $T = 363$ K in a 20 % hydrochloric acid solution for several hours to ensure the complete dissolution of metals into the solution. After acid treatment, the cake was again filtered and washed with distilled water. Samples taken during leaching, the filtrates after leaching and acid treatment, and the wash waters were analyzed for tin ion content using atomic absorption spectrophotometry (AAS) (novAA300, Analytik Jena, Germany). The overall tin recovery (α_{Sn}) was assessed based on its content in the filtrates and wash waters.

Experimental data processing

The degree of tin recovery was calculated, taking into account the volumes of collected samples, using the following formula:

$$\alpha^i_{Sn} = \frac{C^i(V_{\text{initial}} - V_{\text{sample}}(i-1)) + \sum_{1}^{i-1}(C^i V_{\text{sample}})}{G_{Sn}} \cdot 100, \quad (1)$$

where α^i_{Sn} is the degree of tin recovery at the moment of the i-th sample collection, %; C^i is the concentration of tin in the i-th sample, g/dm^3; V_{sample} is the volume of the sample, dm^3; V_{init} is the initial volume of the leaching solution, dm^3; G_{Sn} is the mass of tin in the material sample, g.

Kinetic characteristics (reaction orders and apparent activation energies) were determined by calculating the instantaneous leaching rate at the initial moment of time (υ_0) by constructing tangents to the curves $\alpha_{Sn} = f(\tau)$. The tangents were drawn through the origin of the coordinate system.

Results and discussion

Effect of ultrasonic treatment intensity on tin recovery

The intensity of ultrasonic treatment had varying effects on the systems under investigation (Table 1). In the case of sulphuric acid leaching, increasing the ultrasonic treatment intensity to I = 120 W/cm^2 improved tin recovery by 14.5 %. Further increasing the intensity did not result in a significant rise in tin recovery, which can be attributed to the weakening of cavitation processes due to the excessive growth of cavitation bubble radius [31].

In hydrochloric acid leaching with ultrasonic treatment, tin recovery decreased to 28—29 %, which was comparable to the results of sulphuric acid leaching. The reduction in the efficiency of hydrochloric acid leaching with ultrasonic treatment is likely due to a decrease in hydrogen chloride solubility in the aqueous solution, caused by local overheating at the hot spots formed by the collapse of cavitation bubbles [32].

During ITO leaching in methanesulphonic acid, ultrasonic treatment reduced tin recovery across the entire studied range of ultrasonic intensities to 1.7—4.3 %. This could indicate thermal decomposition of methanesulphonic acid during acoustic cavitation [33]. The slight increase in tin recovery with rising ultrasonic intensity is likely related to the weakening of cavitation processes.

Table 1. **Effect of ultrasonic treatment intensity on tin recovery during leaching in sulphuric, hydrochloric, and methanesulphonic acids (C_{acids} = 0.2 N, T = 333 K, τ = 60 min)**

Таблица 1. Влияние интенсивности УЗО на извлечение олова при выщелачивании в серной, соляной и метансульфоновой кислотах ($C_{\text{кислот}}$ = 0,2 н, T = 333 K, τ = 60 мин)

I, W/cm^2	Tin recovery, %		
	H_2SO_4	HCl	CH_3SO_3H
0	14.9	47.5	5.6
120	29.4	28.0	1.7
180	29.0	28.5	2.8
240	31.6	28.8	2.8
300	31.2	29.3	4.3

Subsequent investigations into the effect of acid concentration and temperature were conducted at a constant ultrasonic intensity of I = 180 W/cm^2.

Effect of acid type and concentration on tin recovery

According to the obtained data (Fig. 2), during ITO leaching without ultrasonic treatment in hydrochloric (Fig. 2, *a*) and sulphuric (Fig. 2, *c*) acid solutions, the most intensive dissolution of tin occurred in the first 20—40 min, after which the process rate significantly decreased due to the reduction in unreacted ITO and the concentration of H$^+$ ions. When leaching in methanesulphonic acid solutions (without ultrasonic treatment), the graphs of $\alpha_{Sn} = f(\tau)$ showed linear behaviour (Fig. 2, *e*) due to the low recovery and the large amount of unreacted tin oxide.

Tin dissolution without ultrasonic treatment was most intensive in hydrochloric acid solutions, where recovery reached 76 % in a 1.0 N HCl solution. In sulphuric and methanesulphonic acid solutions, the maximum tin recovery was 53 % and 48.5 %, respectively (C_{acid} = = 1.0 N). It is clear that the high tin recovery in hydrochloric acid is due to its higher degree of dissociation compared to sulphuric and methanesulphonic acids.

Ultrasonic treatment enhanced tin leaching in sulphuric acid, increasing recovery by 13—30 %, with a maximum of 66% (Fig. 2, *d*). In contrast, acoustic treatment reduced tin recovery in both hydrochloric (Fig. 2, *b*) and methanesulphonic (Fig. 2, *f*) acids. For example,

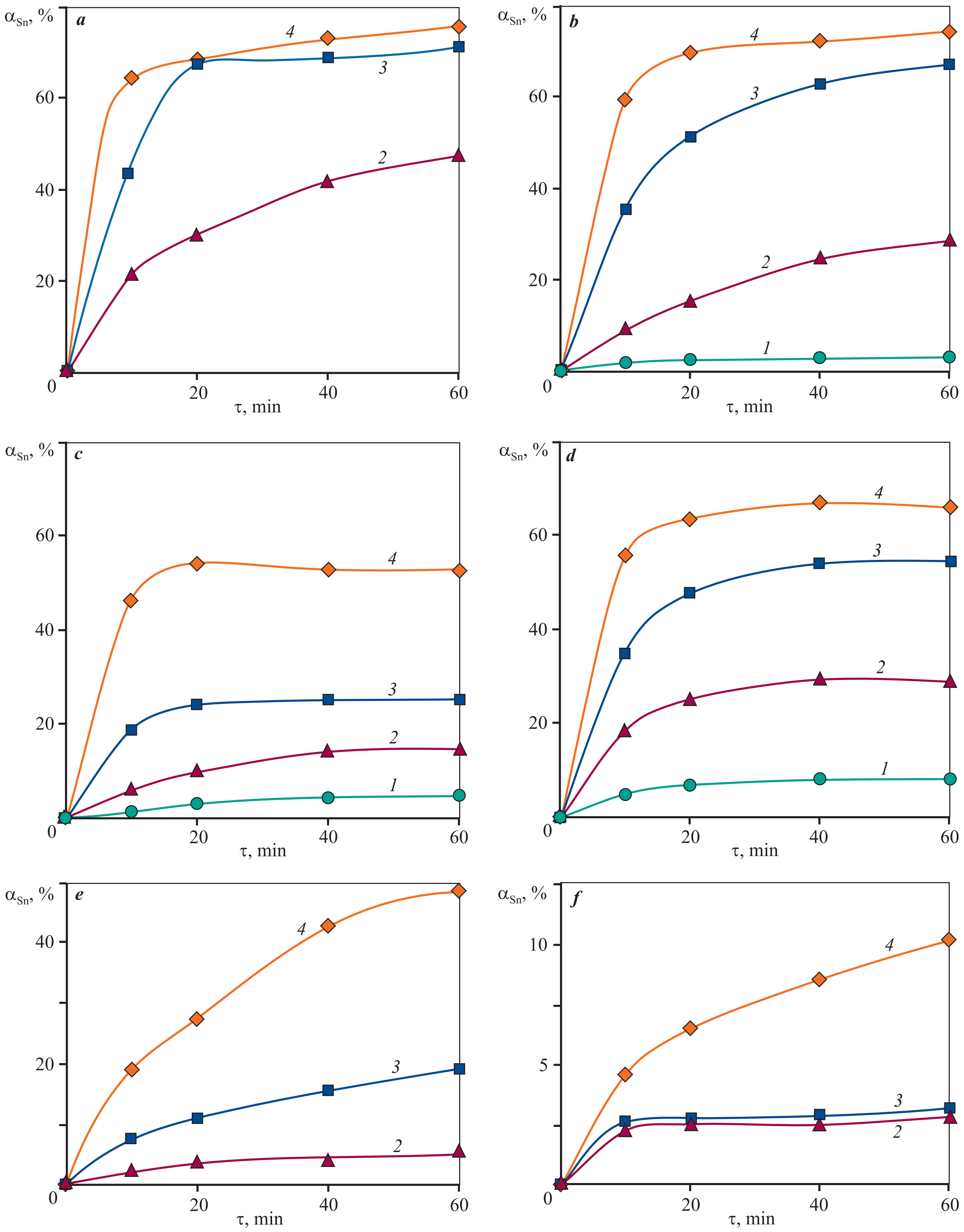


Fig. 2. Effect of leaching duration on tin recovery at various concentrations of hydrochloric (***a***, ***b***), sulphuric (***c***, ***d***) and methanesulphonic (***e***, ***f***) acids without (***a***, ***c***, ***e***) and with (***b***, ***d***, ***f***) ultrasonic treatment

$T = 333$ K; $I = 180$ W/cm^2; C_{acids}, N: 0.1 (*1*), 0.2 (*2*), 0.4 (*3*), and 1.0 (*4*)

Рис. 2. Влияние продолжительности выщелачивания на извлечение олова при различных концентрациях соляной (***a***, ***b***), серной (***c***, ***d***) и метансульфоновой (***e***, ***f***) кислот без использования (***a***, ***c***, ***e***) и с использованием (***b***, ***d***, ***f***) УЗО

$T = 333$ K; $I = 180$ Вт/см^2; $C_{\text{кислот}}$, н: 0,1 (*1*), 0,2 (*2*), 0,4 (*3*) и 1,0 (*4*)

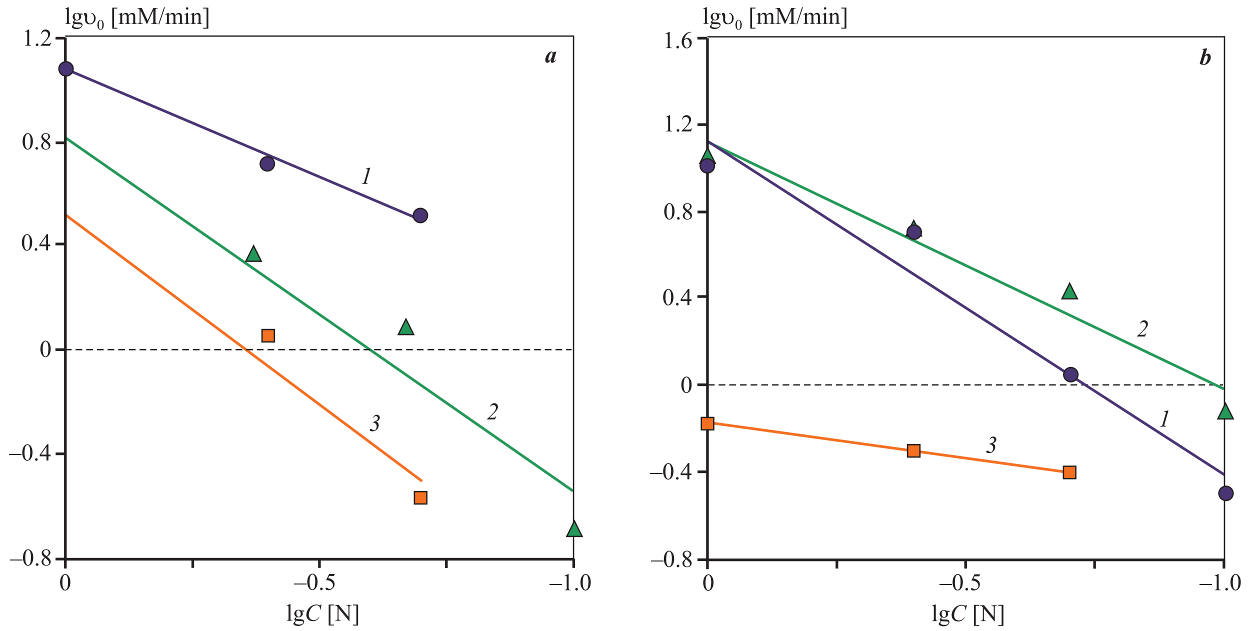


Fig. 3. Evaluation of the partial reaction orders for tin dissolution in hydrochloric (***1***), sulphuric (***2***), and methanesulphonic (***3***) acid solutions without (***a***) and with (***b***) ultrasonic treatment

Рис. 3. Определение частного порядка реакций растворения олова в растворах соляной (***1***), серной (***2***) и метансульфоновой (***3***) кислот без использования (***a***) и с использованием (***b***) УЗО

without ultrasonic treatment, tin recovery in a 0.2 N hydrochloric acid solution reached 48 %, while with ultrasonic treatment, it decreased by 20 %. In methanesulphonic acid, ultrasonic treatment reduced tin recovery by 2 to 6 times, with only 10 % of tin entering the solution after 60 min of leaching in a 1.0 N solution. This significant reduction in tin recovery in hydrochloric and methanesulphonic acids is most likely due to acid decomposition under ultrasonic influence.

The partial reaction orders for tin oxide leaching in the studied acids change significantly under the influence of ultrasound (Fig. 3, Table 2). This effect is especially pronounced in hydrochloric and methanesulphonic acids, which undergo the most significant decomposition under acoustic treatment.

Table 2. **Partial orders of tin oxide leaching reactions by sulphuric, hydrochloric, and methanesulphonic acids**

Таблица 2. Частные порядки реакций выщелачивания оксида олова по серной, соляной и метансульфоновой кислотам

Acid	Reaction order by acids	
	Without ultrasonic treatment	With ultrasonic treatment
HCl	0.8	1.5
H_2SO_4	1.4	1.1
CH_3SO_3H	1.5	0.3

Effect of leaching temperature on tin recovery

Increasing the temperature from 298 to 333 K significantly intensified tin leaching in sulphuric (Fig. 4, *c*) and hydrochloric (Fig. 4, *a*) acid solutions. However, at $T = 353$ K, after 10—20 min of leaching, the concentration of tin ions in the solutions decreased (Fig. 4, *a*, *c*, curve *4*), possibly indicating tin hydrolysis and precipitation at higher temperatures [34].

In the absence of ultrasonic treatment, tin recovery from ITO was highest in hydrochloric acid solutions, reaching 47 % at $T = 333$ K. The application of ultrasound reduced the efficiency of hydrochloric acid leaching compared to experiments conducted without ultrasound: tin recovery decreased by 19—22 % at $T = 333$—353 K (Fig. 4, *b*).

In contrast, sulphuric acid leaching of tin was intensified by ultrasonic treatment across the entire temperature range studied, with tin recovery increasing by 2.0—3.4 times, reaching 30.5 % at $T = 313 \div 333$ K (Fig. 4, *d*). Tin dissolution in methanesulphonic acid was minimal, and tin ions were only detected in solutions after leaching at $T = 333$ K: without ultrasound,

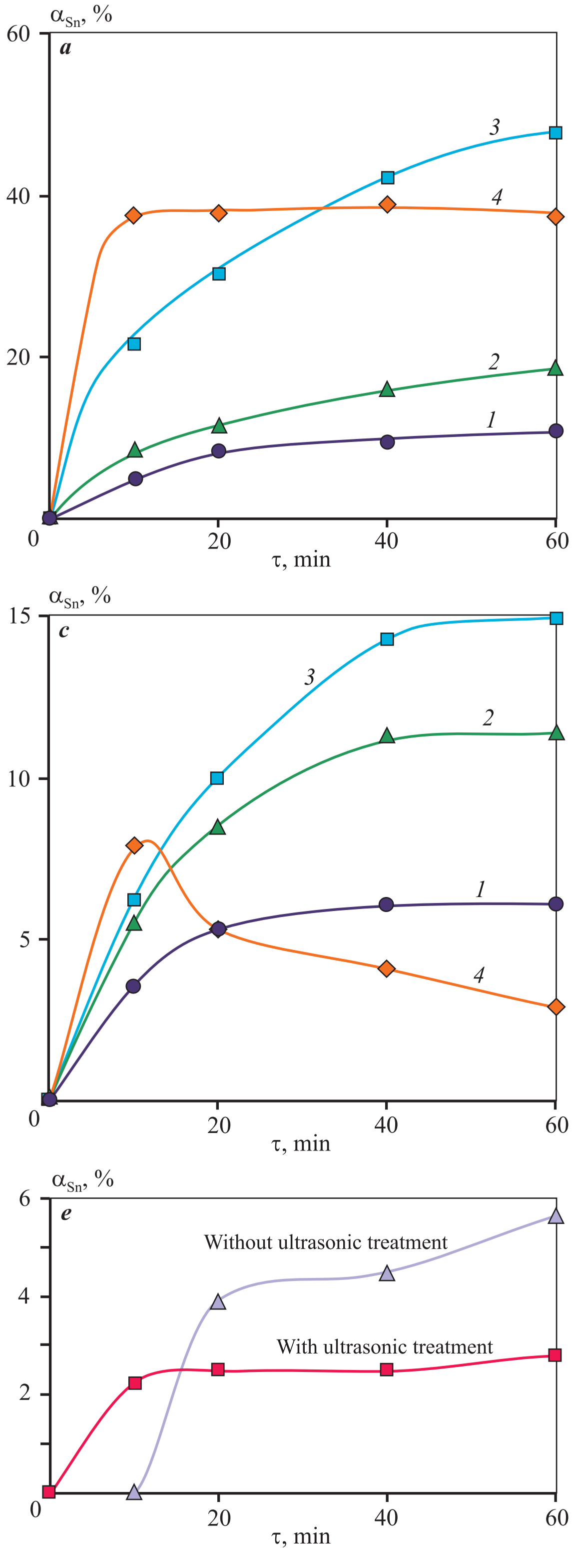


Fig. 4. Effect of leaching duration in hydrochloric (***a***, ***b***), sulphuric (***c***, ***d***), and methanesulphonic (***e***) acids on tin recovery at various process temperatures without (***a***, ***c***) and with (***b***, ***d***) ultrasonic treatment

$C_{\text{acids}} = 0.2$ N; $I = 180$ W/cm^2; T, K: 298 (***1***), 313 (***2***), 333 (***3***), and 353 (***4***)

Рис. 4. Влияние продолжительности выщелачивания в соляной (***a***, ***b***), серной (***c***, ***d***) и метансульфоновой (***e***) кислотах на извлечение олова при различных температурах процесса без использования (***a***, ***c***) и с использованием (***b***, ***d***) УЗО

$C_{\text{кислот}} = 0{,}2$ н; $I = 180$ Вт/см^2; T, K: 298 (***1***), 313 (***2***), 333 (***3***) и 353 (***4***)

$\alpha_{\text{Sn}} = 5.6$ %, and with ultrasound, it dropped to 2.8 % (Fig. 4, *e*).

Based on the obtained kinetic patterns, the apparent activation energy (E_{a}) for the dissolution of tin oxide in hydrochloric and sulphuric acids was calculated (Fig. 5): HCl — 40.4 (22.9) kJ/mol, H_2SO_4 — 4.0 (29.0) kJ/mol (the values in parentheses correspond to leaching with ultrasonic treatment). The E_{a} value and the partial reaction order for tin oxide leaching in hydrochloric acid (without ultrasound) indicate that the process occurs in a kinetic-controlled regime. Under acoustic treatment, the E_{a} value for hydrochloric

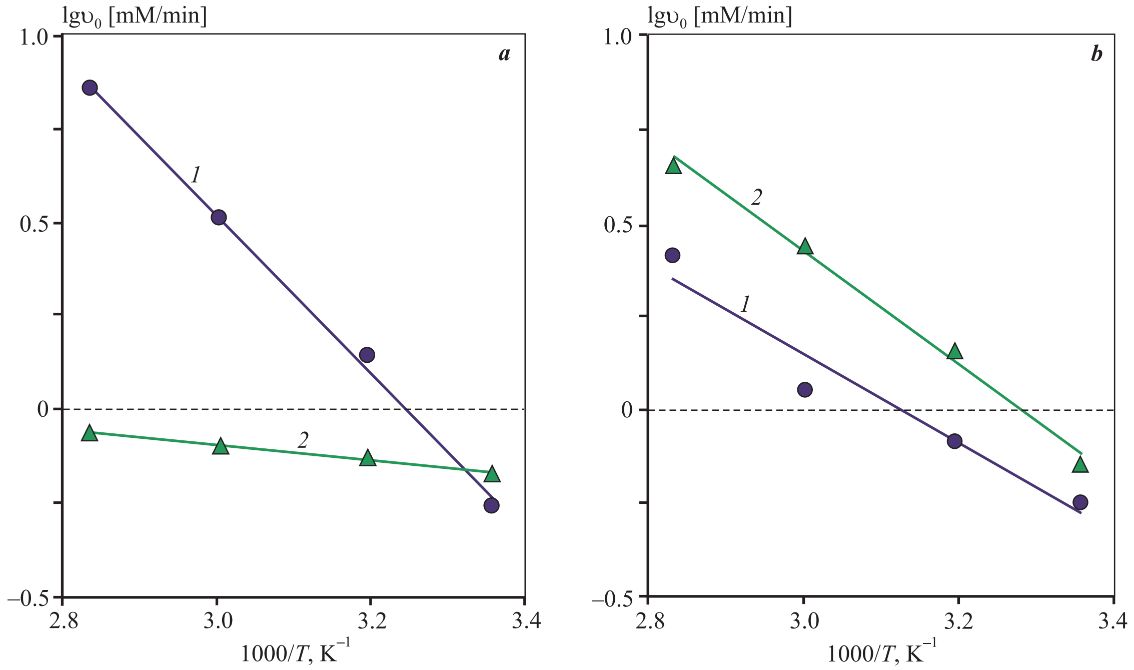


Fig. 5. Evaluation of the apparent activation energy for tin leaching reactions in hydrochloric (***1***) and sulphuric (***2***) acids without (***a***) and with (***b***) ultrasonic treatment

Рис. 5. Определение кажущейся энергии активации реакций выщелачивания олова в соляной (***1***) и серной (***2***) кислотах без использования (***а***) и с использованием (***b***) УЗО

acid leaching of tin oxide decreases, suggesting a shift to a mixed control regime. Sulphuric acid leaching of tin oxide (without ultrasound) likely occurs in a diffusion-controlled regime, while ultrasonic treatment reduces the influence of diffusion factors, leading to a transition to a mixed control regime.

Conclusions

The conducted studies aimed at determining the effects of acid selection, their concentrations, temperature regimes, ultrasonic treatment, and leaching duration of decommissioned displays on leaching performance revealed the following patterns:

1. Ultrasonic treatment intensified the dissolution of tin oxide in 0.2 N sulphuric acid solutions, increasing tin recovery by 14—16 %. However, the application of ultrasound during the leaching of indium tin oxide in hydrochloric and methanesulphonic acids reduced tin recovery to 28 % and 1.7 %, respectively.

2. The maximum tin recovery (76 %) without acoustic treatment was achieved in hydrochloric acid solutions (C_{HCl} = 1.0 N) at T = 333 K over 60 min. With ultrasonic treatment, the highest tin recovery (66 %) was obtained in a 1.0 N sulphuric acid solution at T = 333 K over 60 min.

3. Increasing the leaching temperature significantly enhanced the initial tin dissolution rates. The calculated apparent activation energies for tin leaching in hydrochloric acid indicate a kinetic-controlled process without ultrasonic treatment and a mixed control regime when ultrasound is applied. In sulphuric acid, tin leaching occurs in a diffusion-controlled regime, while ultrasonic treatment transitions the process to a mixed control regime.

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Information about the authors

Elvira B. Kolmachikhina – Cand. Sci. (Eng.), Researcher of the Laboratory of advanced technologies for complex processing of mineral and technogenic raw materials of non-ferrous and ferrous metals, Ural Federal University n.a. the First President of Russia B.N. Eltsin (UrFU).
https://orcid.org/0000-0002-6007-498X
E-mail: e.b.khazieva@urfu.ru

Olga B. Kolmachikhina – Cand. Sci. (Eng.), Associated Professor of the Department of non-ferrous metallurgy of UrFU.
https://orcid.org/0000-0002-7879-8791
E-mail: o.b.kolmachikhina@urfu.ru

Yana A. Yankina – Student of the Department of non-ferrous metallurgy of UrFU.
E-mail: yankina.1999@mail.ru

Zamira M. Golibzoda – Student of the Department of non-ferrous metallurgy of UrFU.
E-mail: golibzoda@mail.ru

Polina A. Brizhevataya – Student of the Department of non-ferrous metallurgy of UrFU.
E-mail: Polina.brizhevataya@mail.ru

Информация об авторах

Эльвира Барыевна Колмачихина – к.т.н., науч. сотрудник лаборатории перспективных технологий комплексной переработки минерального и техногенного сырья цветных и черных металлов Уральского федерального университета им. первого Президента России Б.Н. Ельцина (УрФУ).
https://orcid.org/0000-0002-6007-498X
E-mail: e.b.khazieva@urfu.ru.

Ольга Борисовна Колмачихина – к.т.н., доцент кафедры металлургии цветных металлов (МЦМ) УрФУ.
https://orcid.org/0000-0002-7879-8791
E-mail: o.b.kolmachikhina@urfu.ru

Яна Александровна Янкина – студентка кафедры МЦМ, УрФУ.
E-mail: yankina.1999@mail.ru

Замира Мирзомурод Голибзода – студентка кафедры МЦМ, УрФУ.
E-mail: golibzoda@mail.ru.

Полина Александровна Брижеватая – студентка кафедры МЦМ, УрФУ.
E-mail: Polina.brizhevataya@mail.ru

Daria S. Sedel'nikova – Student of the Department of non-ferrous metallurgy of UrFU.
E-mail: d.s.sedelnikova@mail.ru

Raisa E. Khabibulina – Assistant of the Department of non-ferrous metallurgy of UrFU.
E-mail: raisa.khabibulina@urfu.ru

Дарья Сергеевна Седельникова – студентка кафедры МЦМ, УрФУ.
E-mail: d.s.sedelnikova@mail.ru

Раиса Энверовна Хабибулина – ассистент кафедры МЦМ, УрФУ.
E-mail: raisa.khabibulina@urfu.ru

Contribution of the authors

E.B. Kolmachikhina – defined the objectives of the study, conducted experiments and analyses, and wrote the article.

O.B. Kolmachikhina – contributed to the discussion of the results and co-authored the article.

Ya.A. Yankina – performed experiments, conducted chemical analysis, and participated in the discussion of the results.

Z.M. Golibzoda – carried out experiments and contributed to the discussion of the results.

P.A. Brizhevataya – carried out experiments and contributed to the discussion of the results.

D.S. Sedel'nikova – carried out experiments and contributed to the discussion of the results.

R.E. Khabibulina – carried out experiments and contributed to the discussion of the results.

Вклад авторов

Э.Б. Колмачихина – определение цели работы, проведение экспериментов и анализов, написание статьи.

О.Б. Колмачихина – участие в обсуждении результатов, написание статьи.

Я.А. Янкина – проведение экспериментов и химического анализа, участие в обсуждении результатов.

З.М. Голибзода – проведение экспериментов, участие в обсуждении результатов.

П.А. Брижеватая – проведение экспериментов, участие в обсуждении результатов.

Д.С. Седельникова – проведение экспериментов, участие в обсуждении результатов.

Р.Э. Хабибуллина – проведение экспериментов, участие в обсуждении результатов.

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