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Indium Recycling from Waste Liquid Crystal Displays: Is It Possible?

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Abstract: The utilization of valuable properties of waste and their reuse as raw materials is an imperative of the circular economy. Waste electrical and electronic equipment (WEEE) is a significant source of valuable raw materials, certain metals, and rare earth elements that are the basis for highly sophisticated IT equipment production. It is estimated that the production of WEEE in Europe in 2019 was 16.20 kg/inhabitant, while quantities continue to grow at a rate of 3–4% per year. Waste liquid crystal displays used in televisions, laptops, desktops, and other devices represent a significant share of WEEE and contain 0.12–0.14% of liquid crystals whose main ingredient is indium—tin oxide. In order to investigate and determine the methods and conditions of indium recycling from waste LCDs, laboratory research was conducted. The influence of temperature, particle size, and retention time in different media with and without ultrasound treatment was monitored to provide the efficiency of indium leaching. The analysis of the results showed that 98% indium leaching was achieved with granulation samples of 10 × 10 mm at a temperature 40 °C/40 min in solution H_2O:HCl:HNO_3 = 6:2:1 under ultrasound conditions, while aqueous and alkaline media under the same conditions did not show significant efficiency. This study can be used as a practical reference for the recycling of indium from LCD panels.

Keywords: waste electric and electronic equipment (WEEE); liquid crystal displays (LCDs); liquid crystals; indium recovery; recycling

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1. Introduction

In recent years, waste electrical and electronic equipment (WEEE) has become a global problem that is continuously increasing [1] and is also the fastest-growing single waste stream with an annual growth rate of 3–4%. Such rapid growth can be attributed to the economic development and evolution of electrical and electronic equipment and its increasing integration into everyday activities [2]. According to recent reports from the United Nations (UN), approximately 53.6 million tons of WEEE were produced in 2019, or 7.3 kg of WEEE per capita, a total of 9.2 million tons more than in 2014. It is projected that the amount of WEEE generated in 2030 will exceed 74 million tons [3]. Although the weight of WEEE is not a relevant indicator since its composition changes over the years, the advantage is that a good estimate can be made from the composition of the products placed on the market regarding future WEEE composition.

Liquid crystal display (LCD) waste accounts for a significant share of electronic waste [4]. Today, LCDs are used in televisions, laptops, desktops, and other devices. LCD screens have a short service life period, ranging from 3 to 5 years for personal computer monitors and 1 to 3 years for cell phones [5]. LCD screens are complex in composition and consist of glass (85–87%), polymer membrane (12.7–14%), and liquid crystals (0.12–0.14%). The liquid crystal (LC) consists of glass substrates, liquid crystal, indium tin oxide (ITO), conductive glass, and a black matrix (chromium oxide) [6,7].

LC is placed between two parallel plates (usually glass), with a layer thickness varying between 3 and 5 μm. The glass plates are coated by a thin layer of ITO which has a thickness between 0.1 and 0.3 μm [7].

LCD production consumes approximately 90% of annual indium production [8]. The content of indium in ITO glass powder from waste LCDs can reach 0.0576%, which is more than that in ores containing indium (less than 0.01%). As a result, recycling indium from WEEE is a major source of indium production, but recycling costs limit industrial application [9]. In 2021, a total of 920 tons of indium was produced, and the largest producer of indium is PR China, with a share in total production of 57.6%, followed by South Korea with 21.7%, and Japan and Canada with equal shares of 6.5%. Other indium-producing countries are Belgium, France, Peru, and Russia [10].

Currently, 17% of tin, 21% of silver, and 7% of gold produced annually are used in electronics. In order to move towards a sustainable society and circular economy, WEEE needs to be responsibly processed to recover these critical metals [11].

The European Commission has adopted a circular economy (CE) package that aims to "close" the product life cycle through improved product design, collection, recycling, reproduction, and reuse [12]. Increasing the recycling of end-of-life products reduces the impact on the environment, which is vital for the circular economy [13]. However, research conducted in the work of [14] shows that despite the various policy instruments to boost the CE transition, some gaps require policy attention, i.e., a lack of rules for transparency across supply chains, weak enforcement of waste legislation rules, limited use of circularity criteria in public tenders, and lack of a CE standard.

More than 50% of certain metals, such as iron, zinc, and platinum, are recycled and cover more than 25% of consumption in the European Union (EU). However, when it comes to other metals, particularly those needed for renewable energy sources or advanced technological applications, such as rare earth elements, gallium, or indium, secondary production has only marginal significance. For this reason, much of the potential value for the EU economy is lost, leading to environmental and climate pressures that must be avoided. The European Commission has categorized 30 critical raw materials as key strategic resources, including indium [15].

Indium is a soft, malleable, silvery-white metal that belongs to the group of post-transition metals in the periodic table. Indium is considered a relatively rare element and is not abundant in the Earth's crust, with an average concentration of about 0.1 parts per million (ppm). Indium is primarily obtained as a byproduct during the processing of zinc, lead, tin, and copper ores [16].

At the EU level, research and technological development in relation to the recycling of rare earth elements (REEs) have been supported through the 7th Framework Programme for Research and Technological Development (2207–2013) [17].

If high-content fractions of REEs can be separated out of WEEE streams, the concentration of high-value REEs can be dramatically increased [18].

A large number of end-of-life LCD recycling projects have been developed recently, with the main goal of indium recovery. Most of the methodologies used are based on manual and semi-automatic dismantling, with size reduction, thermal pre-treatments for the removal of organic materials, and hydro-metallurgical processes [19]. The difficulty of LCD disassembly combined with high costs has led to a situation of stockpiling of LCDs at recycling facilities across Europe [20]. Processing technologies, including the recovery of LCs and indium, are still rare or under development according to [7]. The pricing of metals, such as precious metals, is a key indicator of competitiveness in this sector [21,22].

The most commonly reported approach for the extraction of REEs into the aqueous phase is via the use of mineral acids. Several researchers have reported the leaching of REEs from various sources, such as permanent magnets, fluorescent phosphors, catalysts, and other REE-containing wastes using acids such as sulfuric (H_2SO_4), hydrochloric (HCl), and nitric acid (HNO_3) [17].

For waste LCD screens, the work of [23] determined the leaching capacity for indium, arsenic, and antimony. The experimental procedure involved disassembly, shredding material into different fractions (5 × 5 mm, 10 × 10 mm, and 15 × 15 mm), digestion of the shredded material, and evaluating the leaching of these elements. Leaching processes were carried out at different temperatures (20 °C, 50 °C, and 80 °C), using different solid/liquid ratios (S/L) (1:2 and 1:5) and solutions ($HCl:H_2O$ = 3:2; $HCl:HNO_3:H_2O$ = 5:1:4; and $HCl:H_2SO_4:H_2O$ = 2:1:2), to determine the optimal conditions for achieving the maximum leaching capacity. Indium showed the highest leaching percentage (about 60%), while the leaching percentages for arsenic and antimony were very low (0.16% and 0.5%, respectively).

In the research of [24], the authors washed the shredded LCD screens (10 mm) with deionized water at room temperature to remove the organic components. Subsequently, they carried out a two-step leaching process using sulfuric acid at 80 °C for 10 min, resulting in a leaching efficiency of 100%. This washing with deionized water as a pretreatment was necessary to physically remove the organic compounds of the LCDs (rod-shaped molecules containing a benzene ring) [23].

A study by [25] achieved a 90% indium washout efficiency from an uncrushed LCD screen at room temperature using highly concentrated hydrochloric acid (6 M). Meanwhile, in the study of [26], the researchers investigated the use of ultrasonic waves on an uncrushed LCD screen without additional heating using a low concentration of hydrochloric acid (0.8 M). Their method demonstrated a rinsing efficiency of up to 98.8% [26]. In the study of [27], the researchers utilized highly concentrated sulfuric acid ranging from 9 to 18 M. The most effective recycling process for indium was achieved in 3–4 min at a temperature of 60 °C with the sulfuric acid concentration of 18 M utilizing ultrasound. Without the use of ultrasound, the maximum efficiency was reduced to 70% under the same conditions.

The study by [28] involved separating the polarizing film at 230 °C to 240 °C, followed by ultrasonic cleaning at 40 KHz (P = 40 W) using detergents for 10 min. The leaching of indium was carried out in a mixture of concentrated hydrochloric acid (38%) and nitric acid (69%) in the ratio $HCl:HNO_3:H_2O$ = 45:5:50 at a constant temperature of 60 °C for 30 min, resulting in a 92% indium leaching efficiency.

In another study [29], the researchers examined the parameters that affect the amount of dissolved indium in waste LCD screens, such as shredding time, particle size, and duration of acid solution action. The waste LCDs were shredded using high-energy ball milling (HEBM), and the best results for dissolved indium were achieved with a shredding time of 1 min, while longer shredding reduced the amount of indium to about 86%. Acid solutions were used for leaching indium, with a hydrochloric acid solution ($HCl:H_2O$ = 50:50) demonstrating better results within 30 min compared to nitric acid ($HNO_3:H_2O$ = 50:50).

Considering that the shredding of LCD screens significantly affects the leaching capacity of indium [30], the authors investigated two different shredding methods: conventional shredding and electrical disintegration. The conducted research concluded that electrical disintegration was a more efficient method, resulting in a 1.5-times-higher leaching capacity compared to conventional shredding of waste LCDs. The use of electrical disintegration also had a significantly lower environmental impact due to the reduced need for acids as leaching solution.

In further research [31], the recycling of waste LCD screens was studied by combining the process of pyrolysis and immersion of the glass with the ITO layer in sulfuric acid. The polarizing film was treated through pyrolysis, and the indium was leached using 0.6 M H_2SO_4 at 65.6 °C for 42.2 min, resulting in a 100% indium recovery. During their research, the authors identified temperature as the most important parameter compared to other variables.

This paper aims to determine the influence of temperature, particle size, and retention time in different media on the efficiency of leaching indium from waste LCD screens in laboratory conditions in order to research and improve the method of recycling indium from WEEE.

2. Materials and Methods

2.1. Parameters for Conducting Research

To research the efficiency of indium (In) leaching from the LCD screen, three granulations of the LCD screen were used: granulation I, consisting of shredded LCDs smaller than 10 mm; and granulations II and III, consisting of LCD screens cut into squares of 10 × 10 mm and 15 × 15 mm, respectively. For each granulation of the samples, the tests were performed at three temperature and time ratios (20 °C/60 min, 40 °C/40 min, and 60 °C/20 min). The solutions used for leaching were tap water, sodium hydroxide (1 M NaOH), hydrochloric acid solution (H_2O:HCl = 4:1), solution H_2O:HCl:HNO_3 = 6:2:1, and solution H_2O:HCl:H_2SO_4 = 6:2:1. The samples were tested under mechanical mixing and ultrasonic bath conditions.

2.2. Materials

The waste LCD screens used in this research were obtained by disassembling the LCD screens during the mechanical treatment process at the WEEE recycling and recovery plant (Figure 1). Two types of samples were collected and brought to the laboratory, intact LCD screens and LCD screens ground to a size smaller than 10 mm. The granulation of the LCDs to smaller than 10 mm was achieved by shredding in a VM/60 model mill. The intact LCD screens were manually cut into sizes of 10 × 10 mm and 15 × 15 mm.

(**a**) (**b**)

Figure 1. (**a**) Obtained waste LCD screens; (**b**) shredded waste LCD screens.

In the laboratory, the following chemicals were used NaOH (Merck), HCl (37%, AnalaR NORMAPUR), HNO_3 (68%, AnalaR NORMAPUR), H_2SO_4 (98%, AnalaR NORMAPUR), and distilled water to prepare the required solutions. Tap water was used as a leaching medium in the aqueous solution.

2.3. Determination of the Total Concentration of Indium in Samples Dissolved in Aqua Regia

In order to compare the results, an analysis was conducted to determine the total indium concentration in the samples dissolved in the *aqua regia* (Figure 2). *Aqua regia* is a highly corrosive liquid consisting of concentrated nitric and hydrochloric acids, capable of dissolving gold and other resistant substances. The total concentration of elements in the given granulated samples was determined by allowing 10 g of the sample to react with 40 mL of *aqua regia* solution (HCl:HNO_3 = 3:1) for a period of 68 h under controlled conditions. Subsequently, the resulting precipitate was separated from the solution through filtration. The collected filtrate was then analyzed using an atomic spectrophotometer to determine the total concentrations of leached indium. For the granulation of smaller than 10 mm, which exhibited sample heterogeneity, three measurements were performed to determine the total concentration of leached indium, and the mean value was calculated. For the second and third types of sample granulation, one measurement was made for sample uniformity.

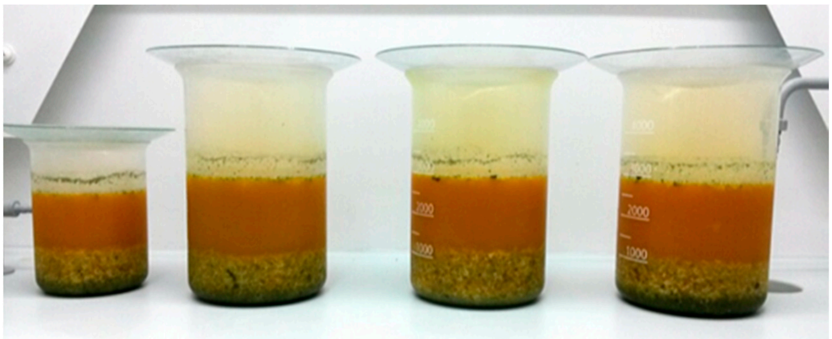

Figure 2. Samples of shredded LCDs in *aqua regia* after 48 h.

2.4. Determination of Indium in Waste LCD by an Ultrasonic Method

Samples were prepared in a 250-mL beaker by adding 10 g of a certain granulation sample and 50 mL of a specific leaching solution. Each prepared sample was weighed individually. In order to prevent solution evaporation during the experiment, the beakers were closed with parafilm. The prepared samples were then treated in an ultrasonic bath at different temperatures and time intervals (20 °C/60 min, 40 °C/40 min, and 60 °C/20 min). For this study, a Bandelin Sonorex Digiplus DL 156 BH ultrasonic bath was used. The ultrasound frequency specified by the manufacturer is 35 kHz, but the experiment was conducted at 50% of the nominal frequency.

After the ultrasonic bath treatment was completed, the beakers containing the treated samples were left to cool at room temperature until completely cooled. This allowed any potential precipitate formed during the treatment to settle at the bottom of the beaker, expediting the filtration process. The samples were filtered using blue strips in a digester to prevent possible evaporation and the spread of unpleasant odors. Tap water was used for treating the samples, while NaOH solution and 0.45-μm filter paper were employed for filtration. The filtrates from the samples were collected in 50-mL glass bottles and labeled uniquely. The analysis of the total concentration of leached indium in the filtered samples was carried out at the Laboratory of Environmental Geochemistry, Faculty of Geotechnical Engineering, using the Hach Lange DR 5000 atomic spectrophotometer using atomic absorption spectroscopy (AAS). The sample is atomized by introducing it into a flame, and a light source is directed through the atomized sample. AAS requires a calibration curve using standard solutions of known indium concentrations.

2.5. Determination of Indium in Waste LCD by Mechanical Mixing

The samples were prepared using the same procedure as the ultrasonic method, but instead of undergoing ultrasonic treatment, they were subjected to mechanical mixing conditions at a speed of 270 rpm. The mixing was carried out using a Phoenix Instruments RSM-03KH mechanical mixer. Filtration of the samples and analysis of the total concentration of leached indium in the filtered samples were performed using the same procedure as employed in the ultrasonic method.

2.6. Concentration of Indium in Samples of Different Granulations Dissolved in Aqua Regia

The total indium concentration was obtained by atomic spectrophotometry of the filtrate obtained by dissolving LCD samples in aqua regia. In order to ensure accurate data comparison and enable mutual comparison, the results were recalculated on the leaching capacity using Equation (1):

$$R = (C \times V)/M \text{ [mg In/kg LCD]} \quad (1)$$

where R represents the leaching capacity of indium [mg In/kg LCD-a], M is the initial mass of the solid sample involved in the leaching process [kg], V is the volume of leaching solution used [L], and C is concentration of the leached element [mg/L].

This recalculation allowed the expression of the results in milligrams of extracted indium per kg of LCD sample. The determined total leaching capacity of indium is presented in Table 1.

Table 1. Total indium concentration and leaching capacity in samples of different granulations dissolved in aqua regia.

	I $Z_{<10\ mm}$	***II*** $Z_{10\times 10\ mm}$	***III*** $Z_{15\times 15\ mm}$
C [mg In/L]	55.44 ± 8.6	55.99 ± 5.37	63.58 ± 2.99
R [mg In/kg LCD]	221.77 ± 2.15	223.96 ± 3.69	254.32 ± 6.13

Table 1 demonstrates that, for granulation I, three leaching experiments were conducted for all tested elements to account for sample non-uniformity. The total leaching capacity value for the first granulation was calculated based on the average of the three measurements given for all elements.

Comparing the achieved leaching capacities in aqua regia, it is evident that the highest leaching capacity was observed in indium from granulation III—254.32 mg In/g LCD. This was slightly lower than the leaching capacity of indium from granulation I—221.77 mg In/g LCD, and from granulation II—223.96 mg In/g LCD.

3. Results and Discussion

3.1. Granulation I =< 10 mm

The capacities of leached indium for granulation I at three different temperatures and time ratios (20 °C/60 min, 40 °C/40 min, and 60 °C/20 min) in solutions B, C, and D using ultrasound (U) and the mechanical mixing method (M) are shown in Figure 3.

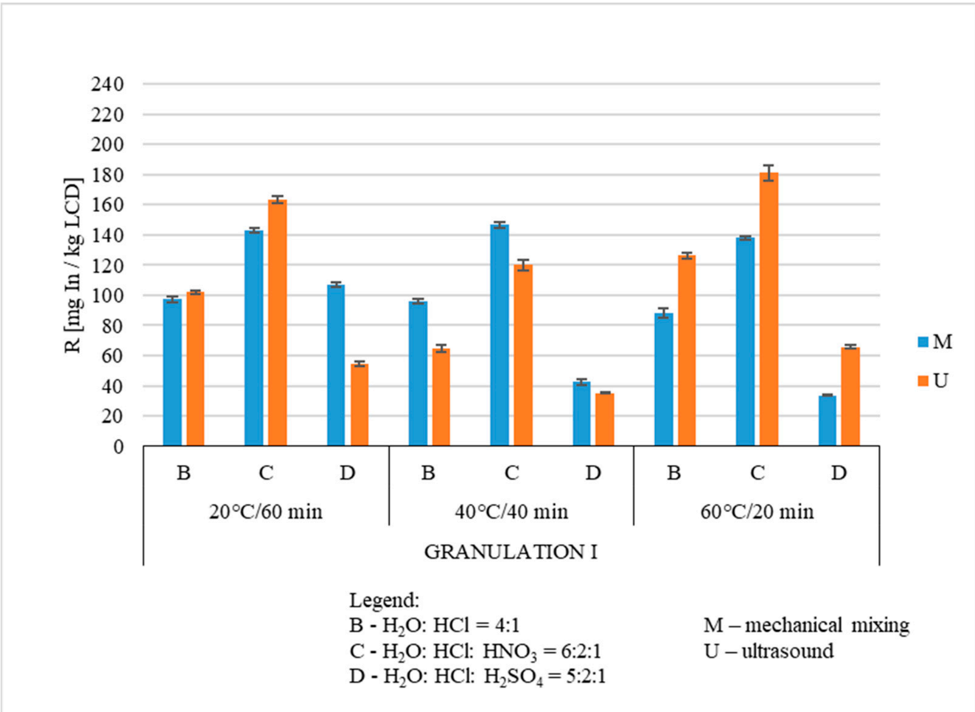

Figure 3. The capacity of leached indium for granulation I.

In granulation I, better results were achieved using solutions B and C for indium leaching compared to solution D. It is notable that solution C consistently yielded the highest amount of leached indium in all three cases when considering solution application. In two cases (20 °C/60 min and 60 °C/20 min), solution C proved to be more efficient. Specifically, the application of solution C at a ratio of 60 °C/20 min in combination with ultrasound resulted in the highest amount of leached indium for granulation I (181.25 mg In/kg LCD). The maximum amount of leached indium for granulation I using mechanical stirring was achieved when the sample was exposed for 40 min in solution C at a temperature of 40 °C. On the other hand, the lowest amount of leached indium for granulation I (33.75 mg In/kg LCD) was obtained when it was subjected to a ratio of 60 °C/20 min in solution D under mechanical mixing conditions. Under ultrasonic conditions, the minimum amount of leached indium for granulation I (35.09 mg In/kg LCD) was obtained from solution D at a ratio of 40 °C/40 min.

3.2. Granulation II = 10 × 10 mm

The leaching capacity of indium for granulation II was evaluated at three different temperatures and time ratios (20 °C/60 min, 40 °C/40 min, and 60 °C/20 min) using solutions B, C, and D. The results obtained through ultrasound (U) and the mechanical mixing method (M) are presented in Figure 4.

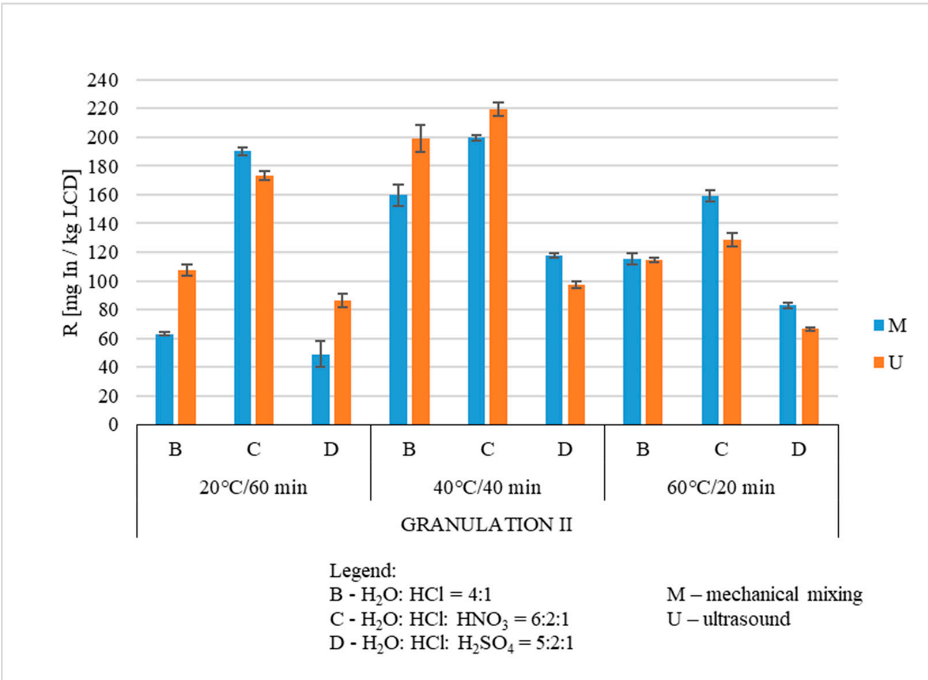

Figure 4. The capacity of leached indium for granulation II.

In granulation II, the highest amount of leached indium was obtained when using solution C compared to leaching with solutions B and D. Notably, the highest leaching capacity of indium (219.30 mg In/kg LCD) was achieved by applying solution C with ultrasound at a ratio of 40 °C/40 min. Under mechanical mixing conditions, using the same ratio and solution, the highest leaching capacity of indium (199.75 mg In/kg LCD) was obtained. Conversely, the minimum amount of leached indium (48.93 mg In/kg LCD) was observed under mechanical stirring conditions in solution D at a ratio of 20 °C/60 min. When comparing mechanical mixing at a ratio of 60 °C/20 min to ultrasound conditions, it was found that a higher leaching capacity of indium was achieved across all solutions. For granulation II, it was observed that the most favorable results in terms of indium leaching capacity, regardless of solution type, were obtained at a ratio of 40 °C/40 min for both mixing methods.

3.3. Granulation III = 15 × 15 mm

The leaching capacity of indium for granulation III was evaluated at three different temperatures and time ratios (20 °C/60 min, 40 °C/40 min, and 60 °C/20 min) using solutions B, C, and D. The results obtained through ultrasound (U) and the mechanical mixing method (M) are presented in Figure 5.

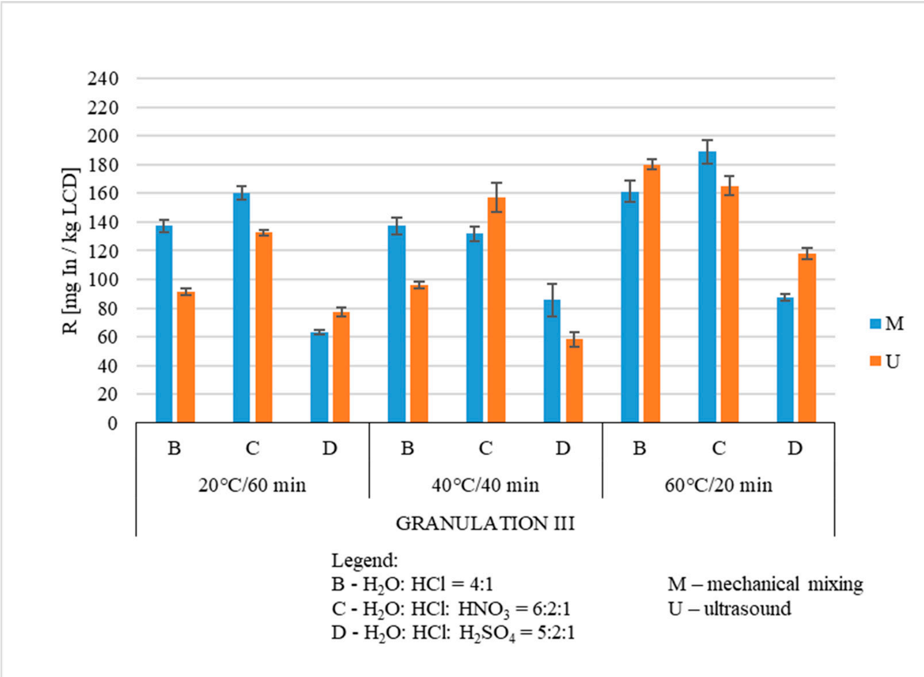

Figure 5. The capacity of leached indium for granulation III.

Similar to granulations I and II, the highest indium leaching capacity was observed using solution C compared to solutions B and D in granulation III as well. The maximum amount of leached indium (188.90 mg In/kg LCD) was achieved using solution C under mechanical mixing conditions at a ratio of 60 °C/20 min. On the other hand, the lowest amount of leached indium in granulation III, across all three temperature-time ratios, was obtained with solution D, regardless of the mixing method. Under ultrasonic conditions, the lowest amount of leached indium (58.35 mg In/kg LCD) was observed at a ratio of 40 °C/40 min. For granulation III, the most favorable results in terms of indium leaching capacity, irrespective of the solution type, were obtained at a ratio of 60 °C/20 min for both mixing methods.

3.4. Efficiency of Indium Leaching from LCDs

Figure 6 presents a graphical display of indium leaching efficiency for all three granulations, considering three different temperature and time ratios (20 °C/60 min, 40 °C/40 min, and 60 °C/20 min) in solutions B, C, and D using ultrasound (U) and the mechanical mixing method (M).

In terms of the solution used for indium leaching, the most favorable results of indium leaching efficiency were obtained using solution C (H_2O:HCl:HNO_3 = 6:2:1). The highest efficiency of 98% was achieved at a ratio of 40 °C/40 min for granulation II. In a previous study [27], the authors also obtained the best results of indium leaching using an H_2O:HCl:HNO_3 solution. On the other hand, leaching in solution D (H_2O:HCl:H_2SO_4 = 5:2:1) resulted in the lowest indium leaching efficiency, as sulfuric acid was found to be less effective in the leaching process, as concluded in studies such as [23,32].

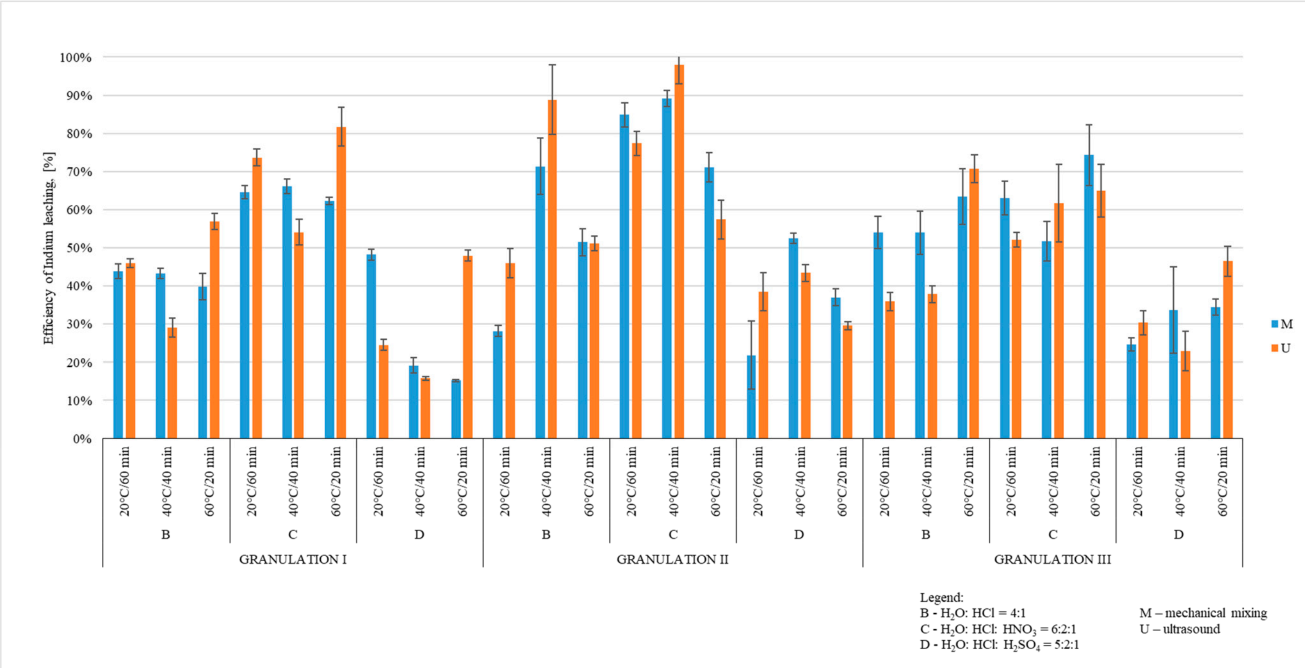

Figure 6. Efficiency of indium leaching depending on method.

Among the different granulations tested, granulation II (10 mm × 10 mm) exhibited the highest indium leaching in all the solutions used. Temperature was identified as a key factor, with higher temperatures and shorter durations yielding better results in terms of indium leaching. Therefore, the leaching efficiency was 98% at a ratio of 40 °C/40 min with ultrasound and slightly lower at 89% with mechanical mixing in solution C for granulation II. Similar conclusions were drawn by the authors in [27,33].

Although the research status in the recovery of REEs from electronic wastes is dynamic and evolving, there are still barriers to widespread WEEE recycling and circular economy diffusion. These barriers include: a lack of awareness that WEEE is a valuable resource, insufficient WEEE collection infrastructure, complex recycling processes, limited resource recovery infrastructure, economic considerations, and policy gaps [34,35].

According to [34], uncertainties and risks in implementing a circular business model arise from the networked nature of the circular economy. Collaboration, communication, and coordination among interdependent stakeholders can be complex, impacting material quantity, quality, and timing. Effective implementation requires a network of information and feedback. The Fourth Industrial Revolution's technological tools provide valuable data, enabling businesses to optimize circular practices and create value. Real-time data collection and information exchange unlock the potential of the circular economy, benefiting a wider range of companies and consumers.

4. Conclusions

Every year, the amount of WEEE generated worldwide is growing at an alarming rate. This increase can be attributed to the global trend of replacing and purchasing new devices, even when the current ones are still functioning. LCD screens, in particular, contain a valuable and scarce element called indium, which is found in nature in extremely limited quantities. The European Commission has categorized indium as a key resource, emphasizing the need for intensive efforts in recycling techniques to recover indium from waste LCD screens. Several recycling techniques have been developed over the years, and some countries have already implemented them extensively to recover indium.

The purpose of this research was to determine the impact of various influential parameters and their synergistic effect on the recovery of indium from waste LCD screens. These parameters included the leaching medium, retention time, temperature, and particle size, all of which play significant roles in the process.

Therefore, this study was conducted to improve the recovery of indium from waste LCD screens, focusing on three different granulations of crushed screens.

The analysis of the results indicated that the most favorable results for indium leaching were achieved with granulation II samples (10 × 10 mm) at a temperature of 40 °C for 40 min in solution C (H_2O:HCl:HNO_3 = 6:2:1) under ultrasonic conditions. This particular combination resulted in an impressive indium leaching efficiency of 98%. It was also observed that solution C (H_2O:HCl:HNO_3 = 6:2:1) consistently yielded the best results for indium leaching across all the granulations tested.

As a suggestion for further research, it would be worthwhile to investigate the concentration of other elements such as copper, aluminum, nickel, chromium, zinc, lead, and arsenic in the collected samples.

The research status in the recovery of REEs from WEEE is dynamic and evolving. However, considering the current market prices, it may not be economically viable to recover these elements from secondary raw materials, as the price of indium in the world is currently around 210–260 USD per kilogram. In a circular economy, the viability of a recycling process largely depends on the market conditions, rather than solely relying on conscientious waste management practices.

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