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**Some innovative applications of reaction-technic and bio-process
engineering in waste management**

Thesis booklet of Doctoral (PhD) dissertation

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1. THE SCIENTIFIC BACKGROUND AND PURPOSE OF THE THESIS

The world's demand for raw materials is constantly growing, but in line with the principles of a circular economy, this growing demand must be met by processing of secondary raw materials. It can therefore be seen that waste recycling, one of the leading research areas of the Institute of Raw Material Preparation and Environmental Processing, will have to play a more important role in the future than perhaps it does today in the educational, scientific and industrial innovation life of the country.

High-volume manufacturing processes generate a large amount of solid wastes, containing valuable minerals and metals, most of which are recoverable. The recovery of these valuable metals would be desirable, as the development of recycling technologies to reduce the volume of primary raw material production is a key element in the circular economy model to be achieved. With the development of the electronics industry, a significant volume of highly valuable secondary raw materials has been generated in waste treatment plants. Electronic waste is characterised by the presence of large quantities of so-called critical elements, the mining of which is both highly polluting and, although they are available in Europe, there are currently no examples of their extraction.

In my PhD dissertation, I investigate possible chemical and biological processes for the dissolution of valuable metals from metal-bearing secondary raw materials, such as electric arc furnace dust (EAF dust), nickel metal hydride (NiMH) batteries and LCD waste.

In my literature search, I reviewed the current practices and research directions for the recycling of the wastes included in my dissertation, focusing on the recovery of target components from the solid phase. I have found that, for base metals, the pyrometallurgical route is preferred, but at least hydrometallurgical treatment is preceded by a thermal pretreatment even if thermal processes are inherently among the most costly processes. The situation is different when the aim is to recover the critical metals such as indium, REEs, gallium, which cannot be separated by conventional metallurgical means and have to be extracted from the slag generated during the process. For e-waste, therefore, the optimal conditions for both chemical and biological leaching are being extensively researched. The results available in the literature show that in most cases conventional solvents (sulphuric acid, hydrochloric acid) are suitable for leaching valuable metals, but selectivity cannot be achieved, even in a few steps, so the solution treatment is usually very complex. A common feature of these studies is that they mostly do not focus on the proper mechanical preparation

of waste with composite composition. Almost without exception, it is uniformly ground to a fine particle size (below 74 μm) before leaching. This is very costly and energy-intensive step, and the reduction of the particle size to such a fine size is not technically sound. Furthermore, the requirement to create a circular economy implies the recovery of all components from the waste. In comparison, very few studies have addressed the recovery of components that can be easily pre-enriched and directly recycled (e.g. plastics, iron).

The aim of my doctoral research was to develop innovative processes and methods to recover target components from different types of secondary raw materials at expected high recovery values, and to investigate the basic phenomena and mechanisms of these processes and methods. Mechanical pre-treatment (comminution, physical enrichment) of primary raw materials is an essential step prior to metallurgical, hydrometallurgical metal recovery. The processes and technological methodologies used for this purpose can be transferred to the processing of secondary raw materials with complex compositions, such as fly ash, electronic and electrical waste, which is the greatest challenge of our time. Accordingly, my further objective is to investigate the possible improvement of the known extraction results by the means of leaching after optimal mechanical preparation. This was investigated for several types of sample materials:

1. In the case of EAF dust, the effect of simultaneous grinding during leaching as a mechanical activation on zinc leaching;
2. Investigation of optimal process parameters for sulphuric acid leaching of NiMH batteries after mechanical preparation for the leaching of REEs; investigation of spontaneous precipitation and hydroxide precipitation from solution;
3. In the context of LCD panel processing, chemical and biological leaching experimentals for the leaching of indium, to determine whether it is necessary to fine-grind the LCD glass prior to leaching.

The aim of my PhD research was also to gain a better understanding of the effect of the different co-substrate constituents on bacterial growth in order to optimise bioleaching. I investigated the applicability and limitations of a new measurement method for *Acidithiobacillus* bacteria using the new generation capacitance measuring Hamilton online cell density meter.

RESEARCH METHOD, EXPERIMENTS

1.1. Equipments

The laboratory leaching experiments were carried out in two different reactors. The leaching experiments to recover the zinc from EAF dust were carried out in a heated stirred ball mill, built at the Department of Process Engineering within the framework of a European R&D project REDILP, to increase the efficiency of the leaching process by using the high grinding energy.

For the chemical and biological leaching experiments of electronic waste, WiseCube WIS-20 incubator shaker was used that can be heated up to 60° C. For the bacterial leaching experiments, sterilisation of the devices and culture medium was performed in a WiseClave autoclave.

1.2. Solvents

In case of the alkaline grinding, a mixture of 25 % ammonium hydroxide (NH₄OH) and ammonium carbonate ((NH₄)₂CO₃) in the appropriate proportions was used as solvent. In the acid chemical leaching experiments, concentrated sulphuric acid (H₂SO₄) and hydrochloric acid (HCl) of analytical reagent grade were used, diluted to the desired concentration with distilled and subsequently deionised water. Two varieties of *Acidithiobacillus* cultivated in Silverman - Lundgren (9K) nutrient solution (*Acidithiobacillus ferridurans* (Karitas) and *Acidithiobacillus ferrooxidans* (Pech)) were used in the bioleaching studies.

1.3. Materials

The simultaneous leaching experiments with the grinding were carried out on fly ash from the Bous steel mill in Germany, with a typical x₅₀ value of 1 µm. The chemical analysis results show that the zinc content of the powder is relatively high, 32%, and the iron content (24%) is also significant. The BET specific surface area measured from the sample is 4.98 m² /g.

The nickel-metal hydride battery sample was sorted, mechanically prepared, shredded, and separated according to physical properties. The resulting fraction with a particle size of less than 2.5 mm, which accounted for almost 70% of the original sample weight, was used for the dissolution experiments. The highest concentration of nickel in the sample is 756 g/kg, while the next highest concentration element is lanthanum, as a rare earth element with 78.8 g/kg.

For the experiments on LCD waste, separate samples were obtained and prepared for chemical and biological leaching tests. The steps of mechanical preparation were the same, the panel was heat treated to remove the foil film and then washed to remove the liquid crystal residue before being shredded in a hammer mill with a 5mm sieve opening. The indium content of the sample ready for dissolution ranged from 155 to 165 mg/kg.

1.4. Analytical methods

For my doctoral thesis, I had to rely on a large number of analytical results from external laboratories. For the analysis of particle size distribution of solid samples, I used a Fritsch Particle Sizer 'analysette 22' followed by a Horiba LA-950 V2 with a measuring range between 10nm and 3mm. X-ray powder diffraction (XRD) studies were performed at the Institute of Mineralogy and Geology, University of Miskolc and Cemkut Ltd. to determine the phase composition. The specific surface area was determined with a BET instrument of the type TRISTAR 3000 at the Department of Metallurgy, University of Miskolc. The SEM results presented in my thesis were measured with the Phenom ProX SEM apparatus at our institute.

Liquid samples were analysed using atomic absorption spectrophotometry and ICP-OES elemental analysis, mostly in external laboratories and at the Institute of Chemistry of the University of Miskolc. For pH and ORP measurements, I used a Hach dual-channel multimeter with a separate "Intellical electrode" set for sterile and bacteria-containing liquids. I was able to use the most probable number method, Bürker chamber counting and the Hamilton cell density monitoring system for the determination of cell counts and bacterial growth.

1.5. Experimental conditions

1.5.1. Zinc recovery by mechanical activation with simultaneous leaching

The aim of my research on electric arc furnace dust was to investigate whether the effectiveness of ammonia-ammonium carbonate solvent dissolution, which has been only partially successful based on literature research and previous institute research, can be increased by mechanical activation when the solid phase has an inherently high ferrite content. Examining the material on which the experiments were based, it was found that the 38% franklinite content, with an adjacent 29% zincite content, together means that 30% of the zinc in the dust is binded into a ferritic structure, making leaching challenging. In

accordance with the research design presented in Figure 2.1, the fly ash sample was prewashed with distilled water at 80 °C prior to the experiments to remove water soluble components (halides, chlorides), the solid residue from the wash was further treated. The parameters varied during the integrated leach-grinding were: initial temperature (30° C, 50° C; 60° C), residence time (30, 60, 90 min) and disc circumferential speed (5, 7.5, 10 m/s). The temperature increased in all cases, but especially during the longer experiments starting from room temperature, due to internal friction of the mill (balls, discs, grains).

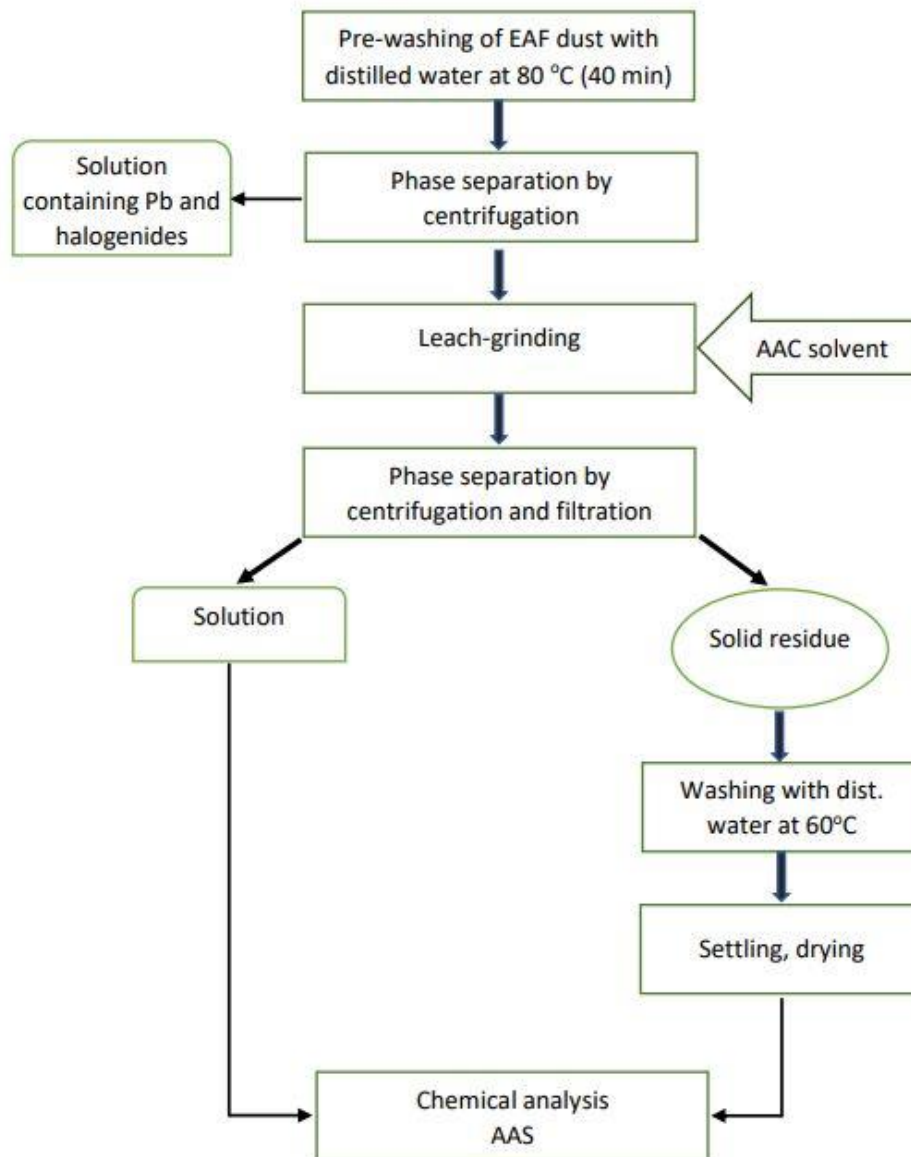


Figure 2.1: Laboratory experiments on EAF dust

1.5.2. Experimental conditions of chemical leaching on nickel-metal hydride type battery

Based on the literature, sulphuric acid is one of the most suitable solvents for the chemical extraction of rare earth elements from NiMH batteries. The dissolution experiments were carried out in a fine fraction of a battery separated on a 2.5 mm sieve in an air flow cabinet under static conditions, i.e. without mechanical stirring, shaking and allowing free escape of gases, with a 2M sulphuric acid solution, after a mechanical preparation developed within the CriticEl project, with a residence time of 2 hours. In further studies, after the gas forming was reduced, a further 1 hour of gentle dissolution with 150rpm stirring was tested in a Wise Cube WIS 20 shaker. In the next phase of the research, two-step leaching experiments were carried out. By this time, as a result of optical microscopy, it was known that the anode material containing my target elements could be cleaned by further classification by size. A significant amount of the interfering elements (plastic fragments, iron and copper wires) could be removed from the material, so the two-step leaching experiments were performed: on a fraction below 0.5 mm further separated by sieving from a sample of $x < 2.5$ mm and on anode material manually separated from NiMH batteries. In the case of the anode, the aim was to see if it was worth to manually separate the anode beforehand. 1M sulphuric acid was used in the first leaching step and 2M sulphuric acid in the second step, with a solid-solvent ratio of 1:10. The experimental flow-sheet is shown in a clear overview in Figure 2.2, from which the origin of the solutions used for the subsequent solution treatment experiments can be traced.

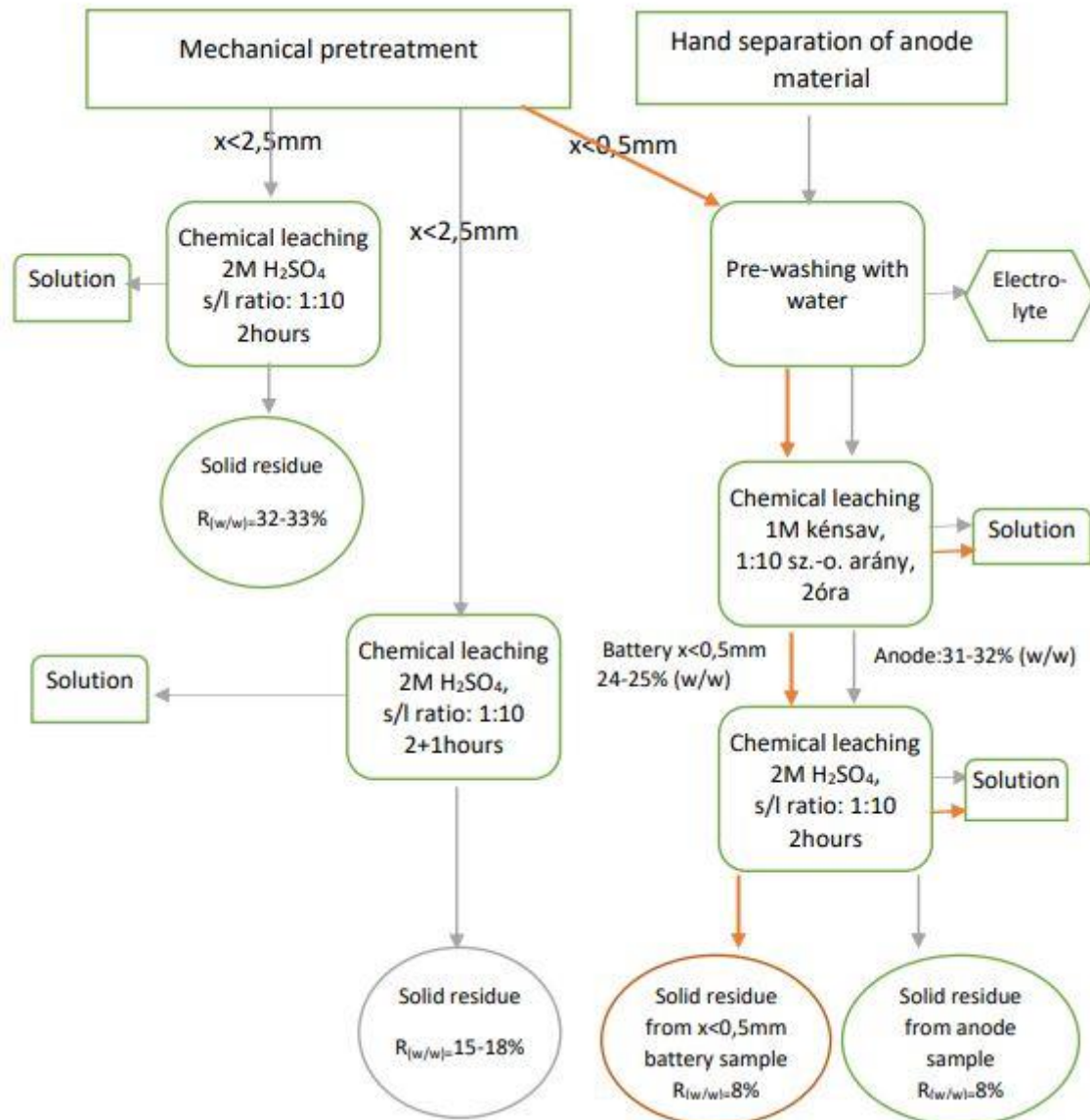


Figure 2.2: Chemical leaching experiments on NiMH batteries and the origin of the liquid phases used for solution handling experiments

1.5.3. LCD chemical leaching experiments

The experiments were carried out on the mechanically pretreated waste LCD panels. The mechanical pretreatment method was developed in the CriticEl project. The parameters of the chemical leaching experiments are summarised in Table 2.1.

1M sulphuric acid and 1M hydrochloric acid were selected as solvents for leaching at 40°C and 55°C, respectively. The parallel experiments carried out with residence times of 1 h and 4 h, mixing was provided by a shaker at 150 rpm. The solid/liquid ratio was 1:1.

Table 2.1: Important parameters of experiments on mechanically prepared LCD glass samples

Particle size	<5 mm		
Initial metal content	In: 155.1 mg/kg, Cu: 89.5 mg/kg, Sn: 152 mg/kg		
Solvent	1M H₂ SO₄ ill. 1M HCl		
Temperature	<i>40°C</i>	<i>55°C</i>	<i>80°C</i>
Solid/liquid ratio	1:1		
Residence time	1h; 4h	1h; 4h	1h; 2h

1.5.4. LCD biosolubilisation experiments conditions

Prior to the systematic biosolubilisation experiments, a preliminary experiment was carried out with a 1:10 solid/solvent ratio using the bacterium *Karitas (Acidithiobacillus ferridurans)*, as no literature data was available on the outcome of the experiment with this bacterium. As a result of the experiment, the indium content of 165 mg/kg in the original solid LCD sample used was reduced to 69.6 mg/kg, which represents a recovery of around 60% and the indium concentration of the solution In content was found to be above 10 mg/l. Further experiments were planned according to this result.

Bacteria were cultured in filter-sterilized “9K” medium containing high (44.2 g/l) ferrous sulphate 7 hydrate at pH 2.5 and inoculated 1:9 ratio for 6 days under static conditions. Subsequently, the experiments with the bacterially inoculated liquid phase were carried out by maintaining a solid/solvent ratio of 10g/200ml at 30° C with continuous agitation for 14 days in a Wise Cube type shaker at 150rpm. In the meantime, pH was measured and samples were taken, during which the amount of sample taken was replenished with 9K nutrient solution. At the start of the biosolubilisation experiments, the pH was 2.3. Experiments were also performed under identical conditions with sterile nutrient solution containing no microorganisms. In addition, for comparison, the growth of bacterial cultures of the same age in the presence of pure nutrient solution or nutrient solution and LCD waste was monitored by the pH change of the system and by using the Hamilton on-line cell monitoring system.

During the biosolubilisation, solution samples for kinetic analysis and the final solutions were analysed by ICP-OES, while the precipitate separated with the solid residue during the process was analysed in some cases after drying also by ICP and Phenom ProX SEM.

2. RESEARCH FINDINGS, THESES

1. thesis

On the basis of the results of my experiments on leaching with mechanical activation for the recovery of zinc from electric arc furnace dust, thermodynamic theoretical calculations, extensive analyses of the phase composition and chemical analysis, it was found that mechano-chemical treatment is suitable for solubilisation of thermodynamically stable ferrites and that supersaturation of the solution leads to the re-formation of ferrites.

2. thesis

Immediate removal of dissolved ions after dissolution of the electric arc furnace dust with ammonia ammonium carbonate is essential to prevent recrystallisation of the ferrites.

Based on these results, I realized that the solubilization of EAF powder should be done in two steps. In the first step of my proposed procedure, zinc is solubilized from the highly soluble zincite in a conventional reactor using ammonia-ammonium carbonate (AAC) solvent. Subsequently, the solid residual zinc containing ferrite is dissolved out in the second step in a suitable stirred ball mill, also with AAC solvent.

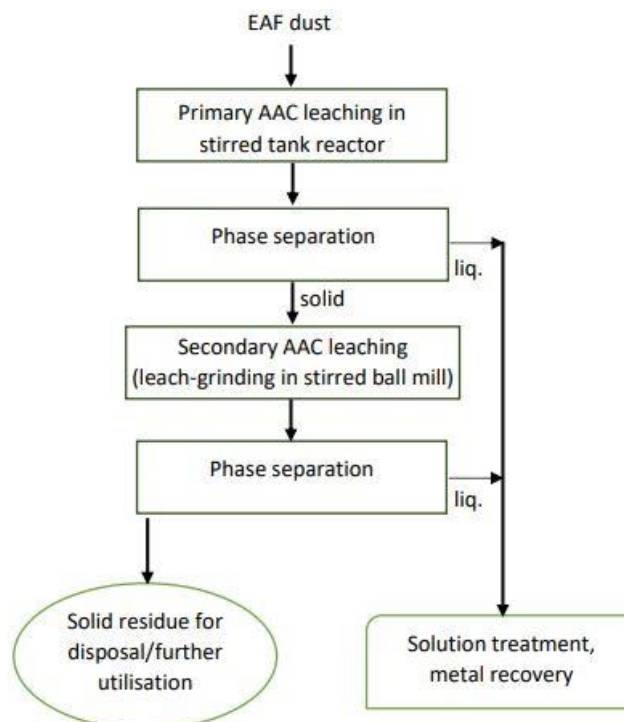


Figure 3.1: Proposed process flow diagram for the extraction of zinc content of EAF powder

3. thesis

Extensive and widespread fundamental process engineering investigation, including the complex examination of physical properties and their distribution, chemical and phase composition, differential solubility, and in the case of e-waste, structural composition, is essential for the development of an efficient waste processing technology. Only such an analysis will allow the establishment of a combined mechanical-chemical-biological technology.

4. thesis

The combined mechanical-chemical-biological technology allows the recovery of components that can be easily removed and recovered by mechanical processes; it also has a beneficial effect on material transfer. After grinding to an optimum degree of liberation, ballast materials can be removed, based on physical property variations, thus increasing the reaction surface area, reducing the chemical demand and avoiding undesired dissolution of certain components (e.g. organic materials, adhesives).

5. thesis

The process I have developed for nickel-metal hydride battery treatment, based on sulphuric acid leaching from the fine fraction enriched during mechanical preparation, is suitable for the recovery of critical elements, in particular rare earth elements, with a solution recovery rate of nearly 100%.

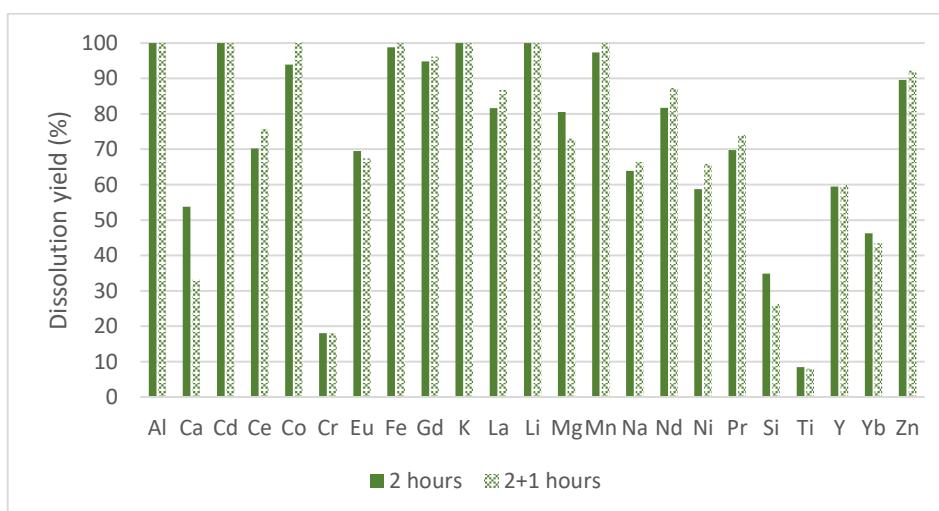


Figure 3.2: Dissolution yield of sulphuric acid leaching of 2 hours and 2+1 hours

6. thesis

Based on the results of the two-step sulphuric acid nickel-metal hydride battery leaching experiments, I found that two-step leaching using a weaker 1M and then a stronger 2M sulphuric acid is not suitable for selective leaching of rare earth metals.

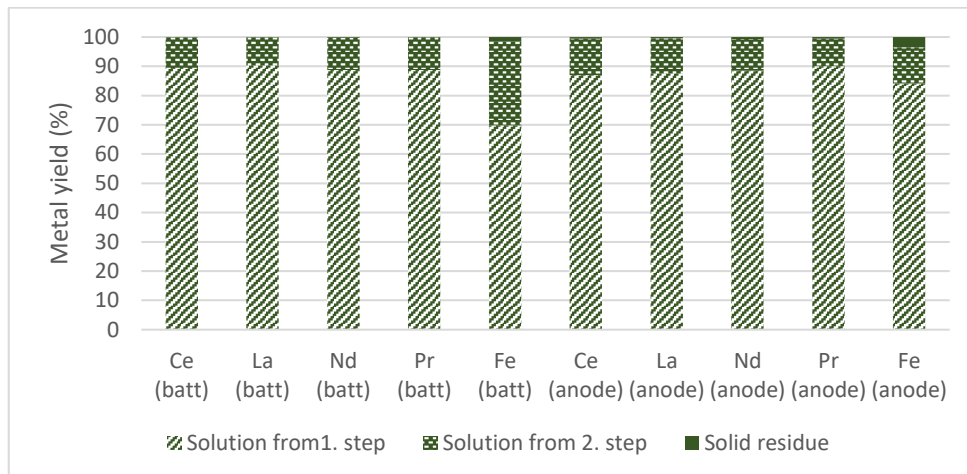


Figure 3.3: Distribution of metal yield values in the two-step leaching stages

7. thesis

I have demonstrated the important fact for the technological design that the manual separation of the anode material have no particular positive effect neither by the aspect of the chemical leaching nor the subsequent solution handling. Considering that the metal yields of mechanically prepared Ni-MH battery samples and manually separated anode samples are very similar. Due to the high nickel content, the violent reaction conditions (gas formation, temperature increase) are the same for the anode sample as for the battery sample. Therefore, manual separation of the anode is not recommended for this reason.

8. thesis

I have found that the precipitation of rare earths with sodium hydroxide after sulphuric acid leaching is a kinetic process, the residence time has the most significant influence on both the recovery and the purity of the precipitate. The recovery value in the precipitation phase is around 90%.

Table 3.1: Metal content of rare earth precipitates formed with NaOH at pH 1.05 according to precipitation time

	Metal content (mg)								
	La	Ce	Nd	Pr	Co	Cd	Ni	Fe	Zn
precipitation t=0	2756	912	371	185	0,94	4,06	6,13	3,11	2,24
precipitation t=1h	2874	955	394	196	0,94	4,2	6,26	1,56	2,82
precipitation t=4h	2909	967	397	194	0,75	4,2	4,85	1,63	1,56
precipitation t=22h	3052	999	414	204	1,91	4,5	13,22	2,73	2,76

When treating the solution obtained from a NiMH battery by sulphuric acid leaching, the aim is twofold. On the one hand, it is necessary to achieve the highest possible efficiency of REE precipitation, and on the other hand, it is important to avoid as much as possible the co-precipitation of other metals and their attachment on the surface of the precipitate. In order to this, I recommend a pH increase to 1.3 for at least 1 hour and gentle mixing for a further 3 hours, according to the results of my experiments. It is worth to separate the spontaneous crystallized solid phase from the sulphuric acid solution of the battery, even after a few hours, before precipitation, so that 7-8% of the REE can be separated in a double sulphate salt form without the addition of any chemical.

Multi-stage precipitation can be used to produce concentrated collective products. It requires a series of redissolution and subsequent solution treatments (e.g. membrane electrolysis, cementation). Accordingly, the proposed process flow diagram is presented in Figure 3.4.

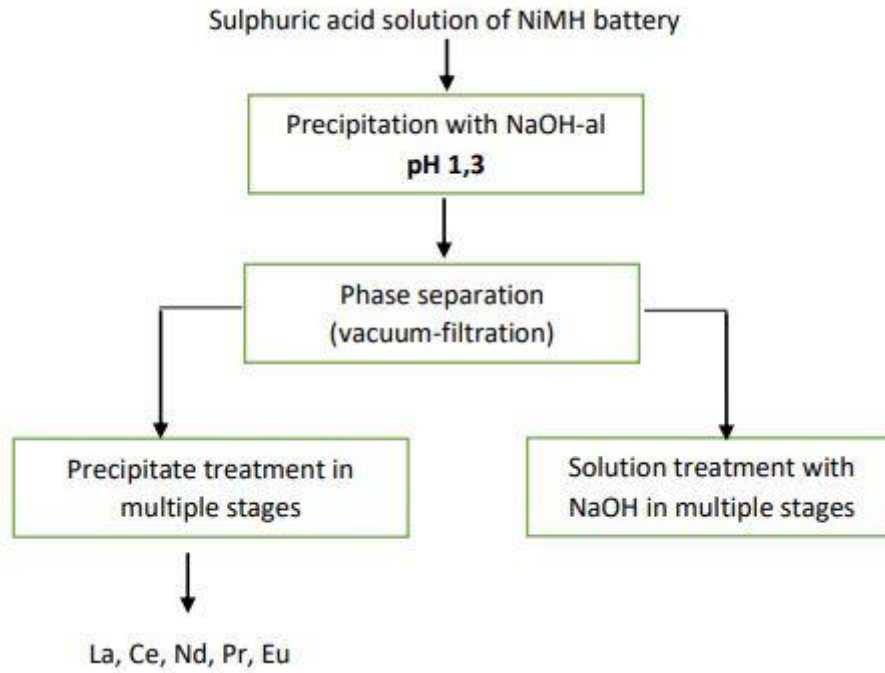


Figure 3.4: Process flow diagram of solution treatment for the recovery of critical metals

9. thesis

I found that indium can be recovered from the LCD panel by a combination of technologies. High concentration of indium can be achieved under optimum conditions (1:1 solid/solvent ratio, 1M hydrochloric acid, 1h residence time, 55° C) with low material flows by hydrochloric acid leaching after mechanical preparation.

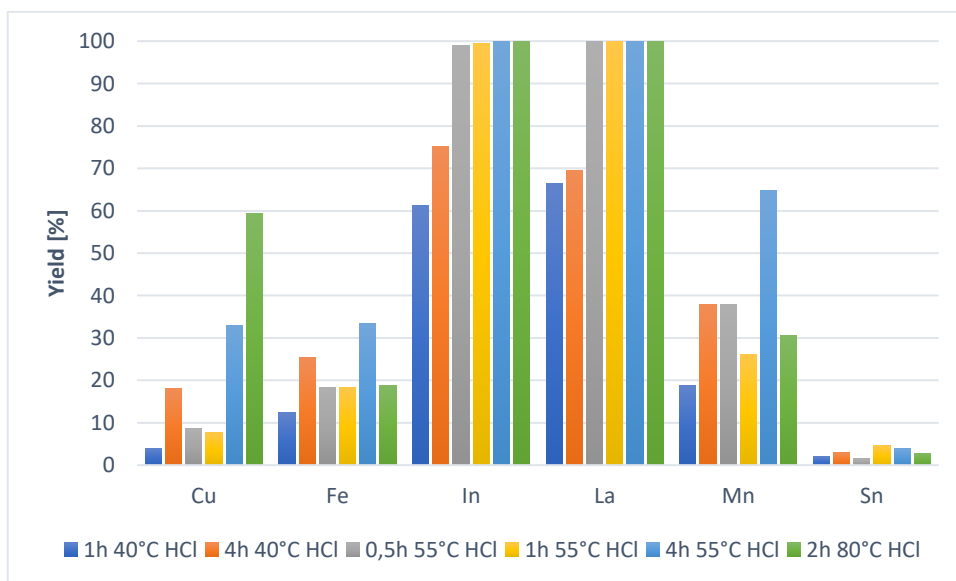


Figure 3.5: Metal recovery values from hydrochloric acid LCD waste leaching experiments

10. thesis

I first demonstrated the ability of the genetically identified bacterial strain *Acidithiobacillus ferridurans* to extract indium from LCD panels under optimal conditions.

Leaching conditions resulting in 60% leaching: mechanically prepared LCD glass, crushed below 5mm, 1:10 solid/solvent ratio, 30° C, 14 days residence time.

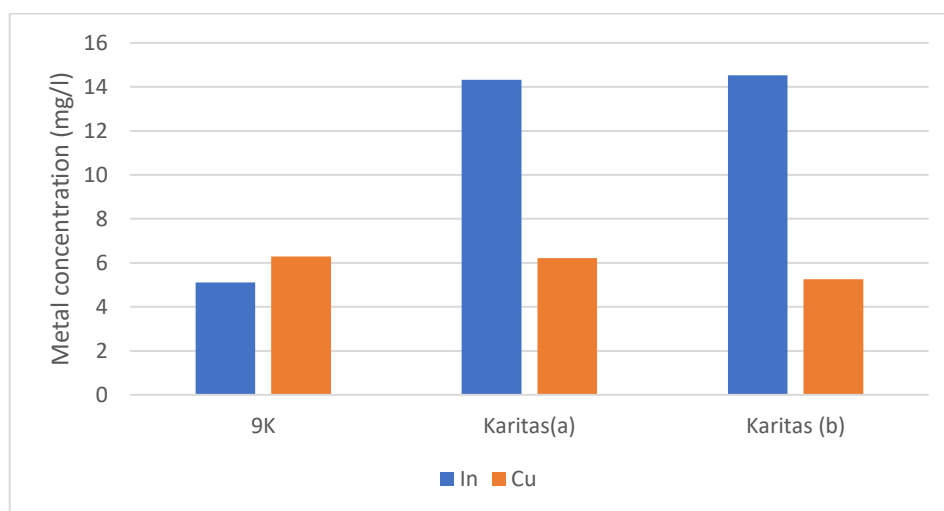


Figure 3.6: Metal concentrations in solutions obtained from solubilisation experiments of the 1-5mm LCD fraction (parallel measurements of Karitas (a) and (b))

11. thesis

The bacterium *Acidithiobacillus* plays an important role in the recovery of the indium content of the ITO (indium-tin-oxide) layer, but this role is not to catalyse the redox reaction but to generate the bio-solvent. This was confirmed by monitoring cell growth. Based on the test results of the chemical and biological solubilisation procedures investigated for the leaching of indium content of LCD, it can be concluded that a lower yield is obtained with a biogenic solvent (up to 60%) than with a chemical solvent (close to 100%).

12. thesis

Based on the construction of a Pourbaix diagram and supported by measurements it was proved, that tin recovery is not possible in a given system. Under the given pH-Eh conditions, the tin oxide phase is stable and the tin content of the LCD panel cannot be recovered by either acid or biological leaching.

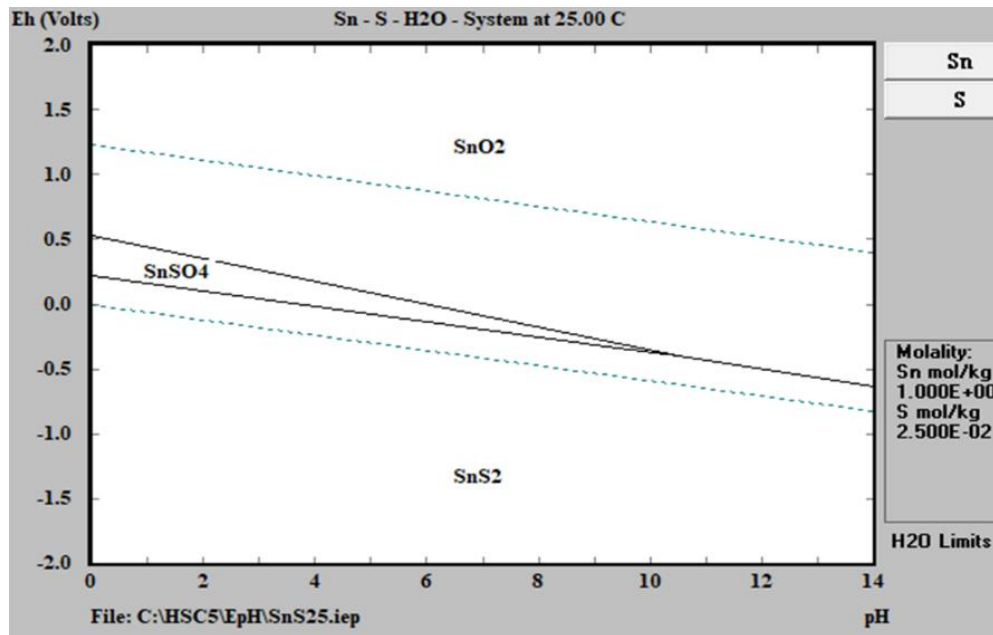


Figure 3.7: Pourbaix diagram for tin oxide

Figure 3.7 shows that the tin oxide phase is stable at the electrode potential value typical of the solutions I have studied, i.e. above 0.5 Volt, so that its dissolution from the ITO layer is not expected.

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The effectiveness of the bioleaching is clearly determined by the successful cultivation of the bacteria, which can be accurately monitored by the new generation Hamilton on-line cell monitoring system, which has not been used for Acidithiobacilli before. The operation of the Incyte sensor is not affected by the fine precipitate, generated during the process, nor is it sensitive to the added waste to be leached when it is LCD glass of maximum 5mm particle size.

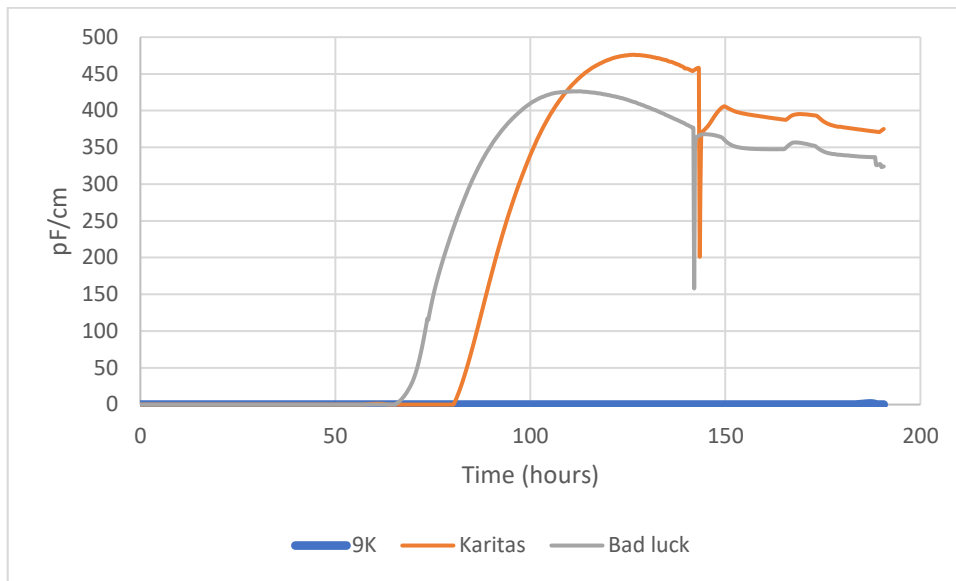


Figure 3.8: Permittivity values measured with the Incyte sensor to monitor changes in live cell count

3. LIST OF PUBLICATIONS ON THE SUBJECT OF THE THESIS

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