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VALUES IN HUNGARIAN BAUXITE RESIDUE: UTILIZATION AS SOIL AMELIORANT AND AS SOURCE OF CRITICAL RAW MATERIALS

Thesis book

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1. Introduction

The scope of this PhD thesis is to establish and provide state-of-the-art technologies that could reuse bauxite residue to highlight its value when being utilised.

Bauxite residue is the slurry by-product generated during the treatment of bauxite ores using the Bayer process to produce alumina. Due to the high volumes generated (~150 million tonnes per annum) as well as the impacts and risks resulting from the disposal, the management of bauxite residue continues to be a global concern. In consequence, there is an immediate need for re-utilization and storage of these residues[1].

Bauxite residue utilization alternatives were classified by Klauber *et al.*[1] into three valuable opportunities studied by several researchers: construction and chemical applications[2], environmental and agronomic applications[3] and metallurgic applications[4].

Wastes and side streams coming from industrial sources for example from mining industry often contain valuable metals. The recovery of these metals from these waste materials may be environmentally favourable and economically viable. Due to the annually generated high bauxite residue amounts it may thus represent an important, untapped secondary source of CRM (and further valuable elements).

Soil degradation and soil contamination have reduced the nutrient content, buffering capacity and detoxification ability of our soils. Thus, protecting soil and preserving its health and overall quality becomes a key goal nowadays. Besides the protection of the soil, another important task for mankind is to manage and utilize waste generated in increasing quantities.

In this PhD research two utilization techniques were evaluated for Hungarian bauxite residue linked to these two issues. In the first-part soil improvement studies were carried out to reveal the opportunity of the utilization of wastes in soil supported by a risk based approach. In the other part a state-of-the-art technology was developed for recovery of critical raw materials (CRM) from bauxite residue.

The main objectives of this thesis are the following:

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The main objectives of this thesis are the following:

1. To predict the amount of bauxite residue that poses no risk to the environment when mixed into the soil.
2. To reveal the beneficial effects of the bauxite residue, as soil ameliorant, on a specific acidic sandy soil in Eastern Hungary.
3. To characterize and evaluate the applicability of the bauxite residue mixed with the agricultural soil (BRSM) as additive to the surface layer of the landfill cover system at a municipal solid waste deposit in Hungary.
4. To create an inventory of valuable elements (CRM including REEs; further valuable metals such as Ni and V) in Hungarian bauxite residue.
5. To develop a technology to recover CRM from Hungarian bauxite residue with combined acid leaching and liquid-liquid extraction (LLE).

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[1] Power *et al.*, 2011. Bauxite residue issue: I. Current Management, disposal, and storage practices. *Hydrometallurgy* 108, 33–45.

[2] Kalkan, E., 2006. Utilization of bauxite residue as a stabilization material for the preparation of clay liners. *Eng. Geol.* 87 (3–4), 220–229.

3 Feigl *et al.*, 2012. Red mud as a chemical stabilizer for soil contaminated with toxic metals. *Water Air Soil Pollut.* 223 (3), 1237–1247.

4 Liu, Z., Li, H., 2015. Metallurgical process for valuable elements recovery from red mud–A review. *Hydrometallurgy* 155, 29–43.

2. Literature review

2.1. General review of bauxite residue and its disposal problems

The aluminium production consists of two key stages. The first is alumina refining (Bayer process), which involves the generation of alumina from bauxite ore, and the second stage is aluminium smelting (Hall–Héroult), which is the process of alumina being transformed into aluminium.

Bauxite residue is a by-product generated from the treatment of bauxite ore with concentrated sodium hydroxide under elevated temperature and pressure in Bayer process in order to dissolve aluminium-bearing minerals (mainly gibbsite, boehmite, diaspore)[5]. An estimated 2.7 billion tons bauxite residue had been produced worldwide until 2007 and that production is increasing by approximately 150 million tons per annum[1]. The production of 1 ton of alumina generates between 0.8 and 1.5 tons of bauxite residue[6]. Spillage of disposed bauxite residue may pose considerable risks to the local environment due to its alkalinity (pH usually ranging from 10 to 13), high sodium concentration (>50 g L^{-1}), fine grained nature (0.7 µm median size of nano-particulate haematite and 1.3 µm median size of cancrinite)[7], and the release of metals and metalloids (e.g., Al, As, Cr, Mo, and V) to the soil–water environment, as demonstrated e.g. following the catastrophic Ajka bauxite residue spill in Hungary, 2010[7]. Therefore, there is an urgent need for new management strategies for bauxite residue.

Topography, availability of land and rainfall are three of the key determinants when choosing the correct method of disposal[1]. Up until the 1970's marine discharge and lagooning were the two methods used, with "dry stacking" (residue is not dry on disposal) and dry cake disposal, the two newest methods of disposal. There is an over 50 years of research and hundreds of publications and patents on what to do with the disposed bauxite residue. All options of bauxite residue reuse are considered, but emphasis is on the few highest volume uses at lowest risk.

There is only one operating alumina plant in Hungary at Ajka with a bauxite residue deposit linked to the plant. In addition, there are two bauxite residue disposal area (Almásfüzitő and Mosonmagyaróvár) associated with decommissioned alumina plants.

Approximately 39.8 million tonnes of bauxite residue in Hungary are stored in bauxite residue lakes (slurries at low densities, 25–30 w/w %), of which 6.4 million tonnes in Mosonmagyaróvár, 14.4 million tonnes in Almásfüzitő and 19.0 million tonnes in Ajka.

2.2. Soil and waste utilization in soil

Soils provide essential ecosystem services for supporting both the ecosystem and the human needs. The intensification and expansion of human activities have placed increasing pressure on land resources, resulting in soil quality deterioration. Different human activities or environmental factors strongly influences soil functions and biodiversity, cause shifts in habitat quality and in substrate availability, resulting in changes in abundance of individual species. The proper soil functioning is a key life support function, so the maintenance of soil quality is critical to environmental sustainability, consequently there is a growing interest in the assessment of the quality and performance of soils that are or may be influenced and degraded by anthropogenic activities. The importance of soil protection among global issues is enhanced because of the impact of the soil degradation on world food security and quality of the environment.

Besides the protection of the soil, another important task for mankind is to manage and utilize waste generated in increasing quantities.

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[5] Gräfe, M., Klauber, C., 2011. Bauxite residue issues: IV. Old obstacles and new pathways for in situ residue bioremediation. *Hydrometallurgy* 108 (1–2), 46–59.

[6] Liu, X., Zhang, N., 2011. Utilization of red mud in cement production: a review. *Waste Manage. Res.* 29(10), 1053–1063.

[7] Mayes *et al.*, 2011. Dispersal and attenuation of trace contaminants downstream of the Ajka bauxite residue (red mud) depository failure, Hungary. Environ. Sci. Technol. 45, 5147-5155.

Linked to these two issues some attempts have been made to use bauxite residue for soil improvement. It has been used in agriculture to increase the phosphorus retention of sandy soil[8] and to increase the low pH of acidic sandy soil[9].

Due to the combined presence of ferric, aluminium, and tectosilicate like compounds in bauxite residue, it is capable of immobilizing toxic metals from polluted soils[10] or removing toxic metals from waste waters[11] or to reduce the leaching of soil nutrients[12].

Risk based approach combined with a value based evaluation of wastes makes possible the matching of certain wastes (e.g. bauxite residue) with degraded or low quality soils to find a technology for utilising the waste in soil improvement. The same waste can pose no risk in one land use, but high risk in another one. Therefore, environmental monitoring during waste utilization in soil, including physico-chemical, biological and environmental toxicity testing is of particular importance.

2.3. Critical raw materials

The search for alternative sources of CRM is urgent as the world economy is largely dependent on their secure supply[13], e.g. for the production of electronic devices and many other technologies. CRM are defined as materials with high supply risk and above average economic importance compared to other raw materials. The supply risk of raw materials is due to the geographic concentration of production concentration [e.g. 99% of heavy rare earth elements (REEs) are produced in China], political and economic stability, potential for substitution and recycling rate[13].

The supply risk can thus be reduced, if so far untapped sources (primary, secondary) can be exploited in the future. Prominent secondary sources are for instance incineration ashes, demolition waste or e-waste. One of so far untapped secondary source for CRM is bauxite residue. Depending on the geological origin of the bauxite ore and operational conditions at which the alumina producers process, bauxite residue can have an elevated content of economically interesting metals[14]. Whereas the major metal content and extractability is well known [iron (Fe), aluminium (Al), titanium (Ti)][15], only a limited number of studies investigated the concentration and extractability of minor constituents such as REEs in bauxite residue. Amongst those studies, almost all exclusively focused on highly abundant REEs like lanthanum (La) and cerium (Ce)[16] and some also included scandium (Sc)[16]. However, recovery of vanadium (V), zirconium (Zr) and gallium (Ga) from bauxite residue is of increasing economic interest. Further CRM may be contained in bauxite residue, but have not been considered so far (e.g. Co, Cr, In).

Though a more complete extraction of the major elements is appealing, processing for recovery of minor CRM from bauxite residue calls certainly for a selective extraction, while leaving Fe and Al substantially undissolved in the bauxite residue.

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[8] Summers *et al.*, 1993. Bauxite residue (red mud) increases phosphorus retention in sandy soil catchment in Western Australia. *Fertil. Res*. 34, 85–94.

[9] Snars *et al.*, 2004. The liming effect of bauxite processing residue (red mud) on sandy soils. *Aust. J. Soil Res*. 42 (3), 321–328.

[10] Gadepalle *et al.*, 2007. Immobilization of heavy metals in soil using natural waste materials for vegetation establishment on contaminated sites. *Soil Sediment Contam*. 16, 233–251.

[11] Garau *et al.*, 2011. Long-term influence of red mud on As mobility and soil physico-chemical and microbial parameters in a polluted sub-acidic soil. *J. Hazard. Mater*. 185, 1241–1248.

[12] Phillips, I.R., 1998. Use of soil amendments to reduce nitrogen, phosphorus and heavy metal availability. *J. Soil Contam*. 7, 191–212.

[13] European Commission, Report on critical raw materials for the EU, 2014. http://ec.europa.eu/growth/sectors/raw-materials/specific-interest/critical/index_en.htm.

[14] Fuller *et al.*, 1982. Reclamation of red mud (bauxite residues) using alkaline-tolerant grasses with organic amendments. *J. Environ. Qual*. 11 (3), 533–539.

[15] Agatzini-Leonardou *et al.*, 2008. Titanium leaching from red mud by diluted sulfuric acid at atmospheric pressure. *J. Haz. Mat*. 157: 579–586 (2008).

[16] Borra *et al.*, 2015. Leaching of rare earths from bauxite residue (red mud). *Miner. Eng*. 76, 20–27.

3. Materials and methods

3.1. Soil improvement study

Linked to waste utilization for soil improvement, a microcosm level laboratory scale study was firstly carried out to understand the effect of bauxite residue on the bauxite residue flooded soil environment. Secondly, a soil improvement microcosm level laboratory scale study was performed to reveal the beneficial effects of bauxite residue as ameliorant of an acidic sandy soil.

The research supported the development of a technology for utilization of BRSM as soil additive. Therefore, a field scale study at a landfill site was carried out to study the beneficial effects of BRSM when applied as landfill surface cover aiming at re-utilizing waste, decreasing cost of waste disposal and providing a value-added product.

Microcosm incubation study – I.

- *Microcosm level in laboratory-scale for 8 months*
- Composition of the microcosms: 5, 10, 20, 30, 40 $^{w}/_{w}$% bauxite residue in untreated soil
- Aim: estimate bauxite residue amount with no adverse effects on the soil as natural habitat
- Questions: How much bauxite residue can be mixed into the soil without adverse effects on soil habitat? What will be the response of the soil microbiota to bauxite residue?

Microcosm incubation study – II.

- *Microcosm level in laboratory-scale for 10 months*
- Composition of the microcosms: 5, 10, 20, 30, 40, 50 $^{w}/_{w}$% bauxite residue in acidic sandy soil
- Aim: reveal the beneficial and soil improvement effects of bauxite residue on a specific acidic sandy soil in Hungary
- Questions: How much bauxite residue can be mixed into the specific acidic sandy soil without adverse effects? What will be the beneficial and soil improvement effects of bauxite residue on specific sandy soil?

Field-scale soil restoration study

- *Field-scale at landfill site for 10 months*
- Composition of the field plots: 5, 10, 20, 50 $^{w}/_{w}$% bauxite residue and soil mixture in subsoil
- Aim: study the beneficial effects of BRSM when applied as landfill surface cover system
- Questions: How much BRSM can be mixed into the landfill surface cover system without adverse effects? Is the BRSM applicable as additive to the surface layer of the landfill?

Figure 1 *The aim and key questions in the first part of the thesis to evaluate the efficiency of a Hungarian bauxite residue as a soil ameliorant*

We monitored the effects of different bauxite residue and bauxite residue mixed with the agricultural soil (BRSM) doses on the soil and the soil habitat with a complex methodology in all experiments that integrates physical, chemical, biological analysis and direct contact ecotoxicity testing with standardized methods as well as newly developed and modified ones.

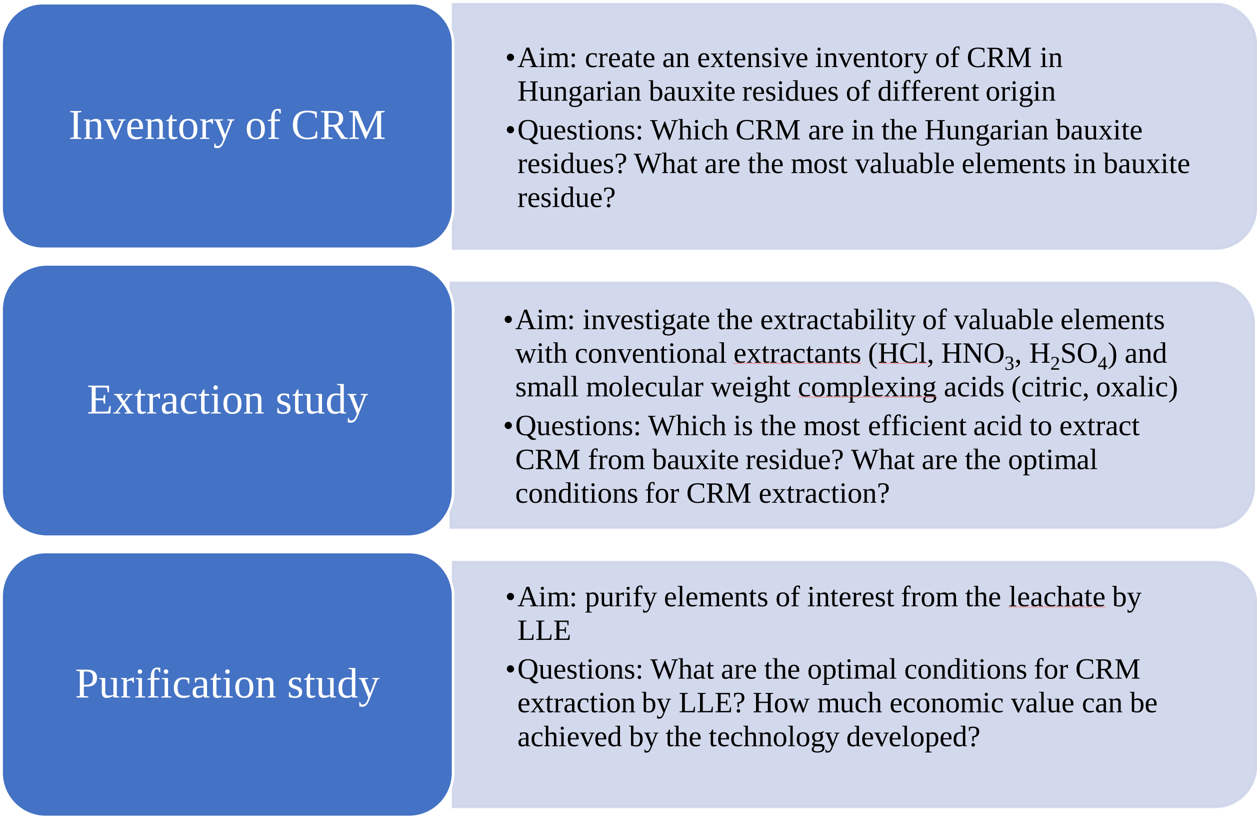
3.2. Recovery study

Since the bauxite residue may contain considerable amounts of CRM, its use as secondary source should be investigated. We propose the development of a technology that does not only recover the economically interesting elements in bauxite residue, yet also provides an option for the mitigation of alkalinity related risks after disposal

For the recovery of valuable elements from Hungarian bauxite residue, an extensive inventory of critical raw materials was created including rare earth elements based on the results of both X-ray fluorescence spectroscopy (XRF) as well as microwave assisted aqua regia digestion with subsequent inductively coupled plasma–mass spectrometry (ICP-MS) analysis.

Next, a number of conventional extracting agents were evaluated for their REE recovery potential. Then, extractability of the REEs by selective acid leaching was also explored in this PhD research.

For comparative evaluation the total economic potential of extracted CRM was calculated as the sum of the products of elemental economic values times the amount extracted.



Inventory of CRM

- Aim: create an extensive inventory of CRM in Hungarian bauxite residues of different origin
- Questions: Which CRM are in the Hungarian bauxite residues? What are the most valuable elements in bauxite residue?

Extraction study

- Aim: investigate the extractability of valuable elements with conventional extractants (HCl, HNO_3, H_2SO_4) and small molecular weight complexing acids (citric, oxalic)
- Questions: Which is the most efficient acid to extract CRM from bauxite residue? What are the optimal conditions for CRM extraction?

Purification study

- Aim: purify elements of interest from the leachate by LLE
- Questions: What are the optimal conditions for CRM extraction by LLE? How much economic value can be achieved by the technology developed?

Figure 2 *The aim and key questions in the second part of the thesis to investigate bauxite residue as a source of CRM*

4. Results

4.1. Soil improvement study

Since bauxite residue (BR) has been successfully used as soil liming material in several applications, we have also found that even the lowest Ajka bauxite residue addition rate (5 $^{w}/_{w}$ %) has shifted the acidic sandy soil (ASS) pH (5.4) to the slightly alkaline domain (7.4) in the microcosm incubation study.

The bauxite residue input to the soil has significantly improved the water holding capacity (WHC) of the sandy soil in both microcosm (Fig. 3) and field experiment. Thus, the WHC improvement is possibly due to the addition of the silt textural class to the sandy soil resulting changes in the porosity and the pore size distribution.

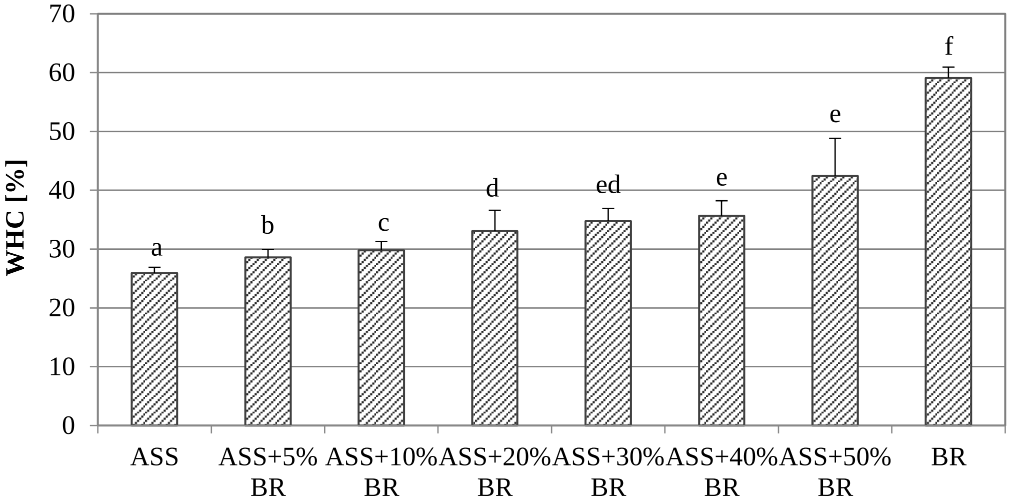


Figure 3 *Effect of bauxite residue on water holding capacity (WHC) in the 10th month. Values followed by the same letter indicate no significant differences at the level of p=0.05 (ASS: acidic sandy soil; BR: bauxite residue)*

The bauxite residue can contain elevated concentrations of potentially toxic metals and metalloids, including As, Cr, Ni and Pb, which were generally associated with residual hard-to-leach fractions of the bauxite residue-affected sediments. The studied bauxite residue had higher total As, Cd, Cr, Ni and Pb content (aqua regia digestible) than the HLV for soil (Hungarian 6/2009 (IV. 14.) KvVM-EüM-FVM decree). The metal content of the soils increased with the bauxite residue loading. The most hazardous metals are As, Cr, Ni and Na based on both microcosms incubation studies. The As, Cr and Ni concentrations exceeded the HLV for soil (As: 15 mg kg^{-1}; Cr: 75 mg kg^{-1}; Ni: 40 mg kg^{-1}) in the microcosms (>5 $^{w}/_{w}$ % and >10 $^{w}/_{w}$ % bauxite residue addition). The As was the single element the concentration of which exceeded the HLV for soil in mixed plots of the field study when the BRSM mixing was higher than 20 $^{w}/_{w}$ %. The Ni concentration also exceeded the limit value but only in the BRSM.

Bauxite residue loading in soil had significantly increased the total and the water soluble Na content compared to the untreated soil due to the high Na content of the bauxite residue. Gruiz et al.[17] recommended Na: 900 mg kg^{-1} concentration as site specific screening value for the soil of the Ajka region. We found that this value had already been exceeded in the 5 $^{w}/_{w}$ % bauxite residue containing soil in both microcosm experiments and in the 10 $^{w}/_{w}$ % BRSM containing soil in the field study.

The leachate pH of the removed BRSM did not differ significantly from subsoil (LQS) and BRSM

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[17] Gruiz *et al.*, 2013. Environmental risk assessment of red mud contaminated soil in Hungary. Proceeding of AquaConsoil 2013 Conference 16–19 April 2013, Theme C: Assessment and monitoring, 2013, paper 2292.

due to the mixing and in addition the bauxite residue contained in the BRSM mixture was carbonated (i.e. neutralised by atmospheric contact). Since the subsoil (LQS) has already had high B, Mo, Na, Ni, Se, and Zn content without being mixed with BRSM, the addition of the BRSM did not influence significantly the metal content of the leachates, except for B and Mo.

The characterization of soil microbiological activity was based on the bacterial cell concentration in the soil in microcosm incubation study I to estimate the bauxite residue amount with no adverse effects on the soil as natural habitat. The Biolog EcoPlate system was applied to investigate the effect of bauxite residue on an acidic sandy soil (ASS, microcosm incubation study II) and of BRSM on a low quality subsoil (LQS, field scale soil restoration study). The Biolog data were evaluated based on various calculated indices, such as average well colour development (AWCD), substrate average well colour development (SAWCD), Shannon diversity index (H), substrate richness (SR) and Shannon evenness (E).

In the microcosm incubation study I average well colour development values indicated that bauxite residue addition at 5 $^{w}/_{w}$ %, 10 $^{w}/_{w}$ % and 20 $^{w}/_{w}$ % increased the activity of the microflora in the acidic sandy soil (ASS) during the short term (5 months), but this effect did not last for 10 months.

According to the Shannon diversity index values the microflora of the low dose BR treated soil (5–20 $^{w}/_{w}$ %) was significantly not different in diversity from the untreated acidic sandy soil over the short term (in the soil in microcosm incubation study II), but by the end of the 10 months experiment bacterial diversity became significantly lower in all bauxite residue treated soils compared to the untreated ASS. The addition of higher bauxite residue (30–50 $^{w}/_{w}$ %) doses should be avoided due to their deteriorating effects on the soil microflora indicated by all calculated indices. In this study, the maximum bauxite residue dose still tolerable by the soil ecosystem in the tested ASS was only 5 $^{w}/_{w}$ %, therefore this amount should not be exceeded.

This is an accordance with the microcosm incubation study I results where the bacterial cell concentration in soil was determined and the number of aerobic heterotrophic living cells did not increase at 5 $^{w}/_{w}$ % bauxite residue dose compared to the untreated soil.

BRSM addition to LQS in field scale soil restoration study at up to 20 $^{w}/_{w}$ % was beneficial for the microbial activity based on AWCD results (Fig. 4).

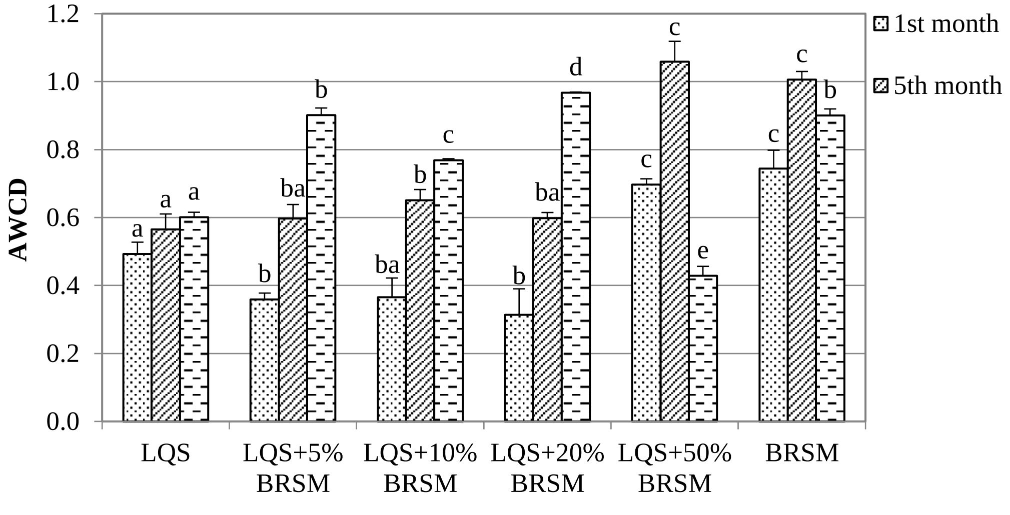

Figure 4 *Average well colour development (AWCD) at 120 h in the samples. Values followed by the same letter indicate no significant differences at the level of p=0.05 (LQS: low quality subsoil; BRSM: bauxite residue-soil mixture)*

In turn 50 $^{w}/_{w}$ % BRSM addition decreased the microbial activity (AWCD values, Fig. 4) and the diversity (based on H). The microbial activity enhancing effect of BRSM addition lasted until the end of the monitored 10 months, as opposed to the short-term effect of bauxite residue in the microcosm incubation study I. The recommended BRSM dose is 20 $^{w}/_{w}$ % based on the biological results.

The potential toxic effect of bauxite residue on test organisms was measured by *A. fischeri* (luminescent bacteria), *T. pyriformis* (unicellular animal), *S. alba* (plant) and *T. aestivum* (plant) and *F. candida* (animal) tests.

The 5 $^{w}/_{w}$ % bauxite residue dose was not toxic to *A. fischeri* in the microcosm incubation study II as compared to the microcosm incubation study I where at 5 $^{w}/_{w}$ % bauxite residue dose the bioluminescence inhibition expressed in Cu equivalent was 1238 mg Cu kg^{-1} soil in the first month (very toxic).

Nevertheless, the 20 $^{w}/_{w}$ % BRSM application was the most toxic to *A. fischeri* causing bioluminescence inhibition in the field plot study, but still in the slightly toxic category. However, the inhibiting effect of the BRSM shows a decreasing trend towards the non-toxic category during the 10 months of the experiment which is in accordance with the results of the microcosm incubation studies.

Studying the effect of BR on plant growth, the 20–40 $^{w}/_{w}$ % bauxite residue dose resulted in significantly smaller *S. alba* root and shoot growth during 8-months-long microcosm incubation study I, compared to the untreated Ajka soil, and to the 5 $^{w}/_{w}$ % and the 10 $^{w}/_{w}$ % bauxite residue treatment. The inhibitory effect of 5 $^{w}/_{w}$ % bauxite residue dose on plants is acceptable as long as it remains under 15%. At higher doses bauxite residue in the ASS affected plant growth negatively. The ED_{50} values showed that the *T. aestivum* plant was more sensitive to bauxite residue compared to *S. alba* in the microcosm experiment. Assessing both the root and the shoot growth results we concluded that 5 $^{w}/_{w}$ % bauxite residue application did not have any significant harmful effect on the soil on the long-term (8 months). Furthermore, the highest inhibition percentage compared to the subsoil (LQS) was observed in case of the *S. alba* root growth in plots containing only BRSM in the field study.

5 $^{w}/_{w}$ % bauxite residue did not inhibit reproduction of *T. pyriformis* as the cell number was similar to the one in the reference sandy soil in the microcosm experiment. The highest inhibition (82%) on the long term was measured at 40 $^{w}/_{w}$ % bauxite residue dose.

The *F. candida* (Collembola) lethality test results of the microcosm incubation study I showed that the bauxite residue loading of the soil did not cause any inhibition in the test organism compared to the untreated soil except for 40 $^{w}/_{w}$ % bauxite residue loading. Similarly, none of the BRSM and subsoil (LQS) mixtures showed inhibition in case of the *F. candida* (Collembola) in the field study compared to the subsoil (LQS) in contrast to the microcosm incubation study I, where the high bauxite residue concentration (> 30 $^{w}/_{w}$ %) in soil was toxic to the test animals.

The microcosm incubation study I showed that 5 w/w % bauxite residue application directly to soil did not have any significant harmful effect on the test organisms, a fact confirmed also by the microcosm incubation study II.

The results of this study underline and demonstrate the need of toxicity test-battery application in relation to hazard and risk assessment, which is able to measure different responses and interactions.

Results of a general technological efficiency assessment carried out by summarizing of significant positive effects of BR and BRSM treatments on soil characteristics are presented in Table 1.

Table 1 *Summary of significant positive effects of various treatments on soil characteristic*

Treatments	**SOIL CHARACTERISTICS**														
	pH	$CaCO_3$	K(A)	Humus	N	P	K	Toxic element	WHC	Microbial activity	*A. fischeri* (bacterium)	*S. alba* (plant)	*T. aestivum* (plant)	*T. pyriformis* (protozoa)	*F. candida* (animal)
	Bauxite residue applied on acidic sandy soil (ASS) as soil ameliorant														
ASS + 5% BR	✓					✓		✓	✓	✓	✓	✓	✓	✓	
ASS + 10% BR	✓	✓	✓			✓		✓	✓	✓	✓	✓			
ASS + 20% BR	✓	✓	✓			✓			✓	✓					
ASS + 30% BR	✓	✓	✓			✓			✓						
ASS + 40% BR	✓	✓	✓			✓			✓						
ASS + 50% BR	✓	✓	✓			✓			✓						
	Bauxite residue-soil mixture (BRSM) as a component of a landfill cover system - mixed with subsoil (LQS)														
LQS+ 5% BRSM	✓							✓	✓	✓	✓	✓	✓		✓
LQS+ 10% BRSM	✓			✓	✓	✓		✓	✓	✓	✓	✓	✓		✓
LQS+ 20% BRSM	✓			✓	✓	✓		✓	✓	✓	✓	✓	✓		✓
LQS+ 50% BRSM	✓			✓	✓	✓			✓		✓				

4.2. Recovery study

The REEs composition of the bauxite residues was enriched in comparison to typical values of <100 ppm to ~500 ppm REE in the bauxites with a total concentration of > 0.11 wt% (1082 mg kg^{-1}) (BR2). From the elements studied, Ga (9.61 US \$ t^{-1}) and REEs (45.25 US \$ ton^{-1}) provided the highest shares of the total economic value so that the further recovery studies focused on both Ga and REEs.

The maximal potential economic value extracted was achieved by HCl (25.48 US \$ t^{-1} for CRM) followed by acids in the order HNO_3 > $C_6H_8O_7$ > H_2SO_4 > $H_2C_2O_4$ [(22.36–9.23 US \$ t^{-1} for CRM (Table 2)]. Based on these findings HCl was, therefore, chosen for further detailed leaching tests.

Table 2 *Extraction efficiencies for leaching bauxite residue with HCl, H_2SO_4, HNO_3, $C_6H_8O_7$ and $H_2C_2O_4$ at (normality = 2; 24h; 60 °C; 100 g L^{-1} slurry concentration)*

Acid	**Final pH**	**Recovery efficiency***					
		Fe	**Al**	**Ga**	**La**	**Ce**	**Nd**
HCl	0.19	3.0 ± 0.1% (6529 mg kg^{-1}) a	94.6 ± 4.1% (47508 mg kg^{-1}) a	44.3 ± 2.3% (11.8 mg kg^{-1}) b	51.0 ± 2.6% (84.5 mg kg^{-1}) a	39.6 ± 1.4% (170 mg kg^{-1}) a	58.5 ± 2.1% (88.4 mg kg^{-1}) a
H_2SO_4	0.49	1.9 ± 0.1% (4130 mg kg^{-1}) c	84.1 ± 4.8% (47453 mg kg^{-1}) a	31.9 ± 1.6% (8.50 mg kg^{-1}) c	28.9 ± 1.7% (47.8 mg kg^{-1}) c	22.9 ± 0.9% (98.4 mg kg^{-1}) b	27.1 ± 1.4% (41.0 mg kg^{-1}) c
HNO_3	0.22	1.2 ± 0.03% (2502 mg kg^{-1}) d	91.4 ± 2.0% (51541 mg kg^{-1}) a	42.4 ± 0.5% (11.3 mg kg^{-1}) b	39.3 ± 0.5% (65.0 mg kg^{-1}) b	24.0 ± 0.6% (103 mg kg^{-1}) b	49.6 ± 0.6% (75.0 mg kg^{-1}) b
$C_6H_8O_7$	2.61	2.8 ± 0.1% (6083 mg kg^{-1}) b	80.2 ± 3.0% (45230 mg kg^{-1}) a	30.6 ± 0.5% (8.15 mg kg^{-1}) c	38.2 ± 1.0% (63.3 mg kg^{-1}) b	24.0 ± 0.6% (103 mg kg^{-1}) b	48.6 ± 2.0% (73.4 mg kg^{-1}) b
$H_2C_2O_4$	0.7	2.0 ± 0.1% (4389 mg kg^{-1}) c	9.0 ± 0.2% (5054 mg kg^{-1}) b	59.6 ± 4.9% (15.9 mg kg^{-1}) a	<LOD d	<LOD d	<LOD d

Values followed by the same letters, indicate no significant differences at the level of $p<0.05$. Elemental analysis: according to Chap. 3.2.3 by ICP-MS.

*Based on aqua regia accessible content.

Increasing HCl concentrations from 0.5 to 6 M led to considerably increased extraction efficiencies of REE (La, Ce, Nd) and Ga. The best results regarding contact time were achieved at 3 hours, showing high Ga (63%), La (67%), Ce (54%) and Nd (69%) extraction efficiency, yet comparatively low Fe extraction efficiency (21%). Besides, the increase of temperature and slurry concentration had only a minor effect on the latter elements.

Visualizing complex interactions between extraction parameters may allow for a careful interpretation of processes underlying extraction. Multitude of different parameters (acid concentration, temperature, time, slurry concentration) makes it difficult to compare. Here the systematic model shows the two-factor and non-linear (quadratic) interactions.

Optimal conditions maximizing the economic potential were predicted for 5.60 M HCl, 24 h contact time, 73.36 °C, and 100 g L^{-1} slurry concentration.

Indeed, experimentally determined economic potential corresponded well (96 % of predicted) to the predictions, allowing a maximum recovery of 40.95 ± 0.90 US $ t^{-1}.

Due to the fact that Fe and Al are the most abundant elements in bauxite residues in general, their removal is a crucial factor regarding selective recovery of other elements. Here, we could show efficient separation by precipitation (Fe) or by removal with the raffinate (Al) of the LLE. A good selectivity for REEs towards Fe was achieved using simple precipitation, since 87% of Fe precipitated from the leachate at pH 3 (Fig. 5). Next to Fe, a major proportion of Al (33%) was removed as well, whereas most REEs (79%) could be further purified by subsequent LLE. Here, Al remained to large extent in the raffinate (59%).

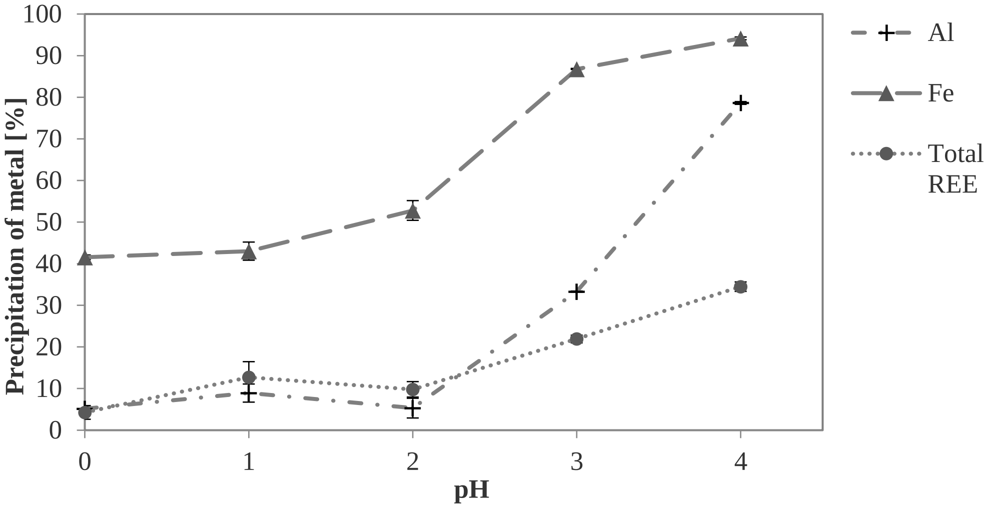

Figure 5 *Precipitation of Al, Fe and total REEs with 1M NaOH at different pH.*

General trends of the influence of LLE parameters on REE extraction / selectivity have been observed: increasing extraction of REEs with increasing D2EHPA concentrations and with increasing LLE organic/aqueous ratio. For stripping, higher HCl concentration lead to higher stripping efficiencies.

With the whole process the maximum economic value of $17.18 US $ t^{-1} was recovered with the optimal conditions predicted by the model regarding recovery of maximal economic value of CRMs were 0.1, 0.76 M, 0.67, and 4 M for organic/aqueous ratio during extraction, D2EHPA concentration in kerosene, organic/aqueous ratio during stripping, and HCl concentration, respectively. CRM prices may strongly rise in the future due to increasing demand and high supply risk, making the proposed processes even more economically attractive. The prediction model was accurate, since the experimentally determined economic potential corresponded well (88%) to the predictions and allowed a maximum recovery of 17.18 ± 0.59 US $ t^{-1}.

Table 3 *Achieved economic value of metal(loid)s recovered from 1 t bauxite residue bauxite residue with 5.37 M (S/L 100; 73.21 °C; 23.24h)*

Element	Aqua regia accessible [g t^{-1}]	HCl leachate [g t^{-1}]	Supernatant [g t^{-1}]	LLE organic solvent phase [g t^{-1}]	Stripping media [g t^{-1}]	Price* [US $ t^{-1}]	Overall economic value [US $ t^{-1}]	Economic value in HCl leachate [US $ t^{-1}]	Economic value in Supernatant [US $ t^{-1}]	Economic value in LLE organic solvent phase [US $ t^{-1}]	Economic value in stripping media [US $ t^{-1}]
Al	56401 ± 1542	54735 ± 1473	36505 ± 1721	2952 ± 147	3001 ± 151	2,092	117.99	114.51	76.37	6.68	6.28
Ce	430 ± 2.7	240 ± 5.3	205 ± 1.3	196 ± 4.1	177 ± 9.2	4,500	1.93	1.08	0.92	n.a.	n.a.
Co	59.5 ± 0.5	47.4 ± 0.9	41.3 ± 1.4	–	–	31,747	1.89	1.50	1.31	0.00	0.00
Cr	646 ± 4.2	614 ± 16.8	45.4 ± 0.1	35.6 ± 1.8	33.4 ± 1.2	10,866	7.02	6.67	0.49	0.39	0.36
Dy	21.2 ± 0.1	14.2 ± 0.2	10.5 ± 0.4	10.5 ± 0.4	9.96 ± 0.02	685,000	14.52	9.73	7.19	7.19	6.82
Er	12.0 ± 0.1	7.36 ± 0.1	6.32 ± 0.05	6.32 ± 0.05	4.17 ± 0.5	705,000	8.46	5.19	4.46	4.46	2.94
Eu	6.07 ± 0.01	3.59 ± 0.1	3.06 ± 0.1	3.06 ± 0.1	2.86 ± 0.2	12,500	0.08	0.04	0.04	0.04	0.04
Fe	214175 ± 4365	164915 ± 2781	21765 ± 147	20226 ± 834	14566 ± 519	0,068	0.01	0.01	n.a.	0.00	0.00
Ga	26.6 ± 1.2	17.6 ± 0.5	5.12 ± 0.	1.99 ± 0.1	1.83 ± 0.2	362,000	9.63	6.37	1.85	0.72	0.66
Gd	26.6 ± 0.4	18.3 ± 0.1	14.9 ± 0.4	14.9 ± 0.4	12.7 ± 1.3	12,500	0.33	0.23	0.19	0.19	0.16
Ho	4.15 ± 0.1	–	–	–	–	12,500	0.05	n.a.	n.a.	n.a.	n.a.
In	0.555 ± 0.01	–	–	–	–	735,000	0.41	n.a.	n.a.	n.a.	n.a.
La	166 ± 1.4	120 ± 2.0	103 ± 0.8	86.4 ± 0.01	71.4 ± 2.1	5,000	0.83	0.60	0.51	0.43	0.36
Lu	1.72 ± 0.2	–	–	–	–	12,500	0.02	n.a.	n.a.	n.a.	n.a.
Nd	151 ± 1.1	106 ± 1.6	93.0 ± 0.3	93.0 ± 0.3	79.0 ± 5.7	58,000	8.76	6.15	5.39	5.39	4.58
Pr	39.2 ± 0.3	26.5 ± 0.6	24.0 ± 0.2	24.0 ± 0.2	19.7 ± 1.2	12,500	0.49	0.33	0.30	0.30	0.25
Sc	80.0 ± 2.6	68.7 ± 1.2	14.8 ± 0.6	14.8 ± 0.6	–	12,500	1.00	0.86	0.18	0.18	n.a.
Sm	27.9 ± 2.4	20.6 ± 0.6	16.1 ± 0.4	16.1 ± 0.4	15.7 ± 1.2	12,500	0.35	0.26	0.20	0.20	0.20
Tb	3.69 ± 0.3	–	–	–	–	615,000	2.27	n.a.	n.a.	n.a.	n.a.
Tm	1.78 ± 0.03	–	–	–	–	12,500	0.02	n.a.	n.a.	n.a.	n.a.
Y	100 ± 9.4	35.9 ± 0.6	–	–	–	60,000	6.00	2.15	n.a.	n.a.	n.a.
Yb	10.8 ± 1.2	7.13 ± 0.2	2.74 ± 0.1	2.74 ± 0.1	1.72 ± 0.06	12,500	0.13	0.09	0.03	0.03	0.02
Total							182.20	155.77	99.45	27.08	23.46
Total CRM							64.20	41.26	23.08	20.41	17.18
Total REE							45.25	26.71	19.42	19.30	16.16

Elemental analysis: according to Chap. 3.2.3 by ICP-MS.

*Values from U.S. Geological Survey (USGS, 2015).

5. New scientific findings of the dissertation – thesis points

Evaluation the bauxite residue as a soil ameliorant:

1. I proved that bauxite residue from Ajka (Hungary) spilled on the soil surface could be mixed at up to 5 $^{w}/_{w}$ % into a sandy soil without any long-term adverse effect on the soil habitat based on the results of a complex methodology that integrates physical, chemical, biochemical analysis and ecotoxicity testing. (VI)
2. I determined based on the applied complex monitoring methodology that the bauxite residue produced in Hungary and stored in the local bauxite residue disposal area was suitable for the improvement of the pH, the $CaCO_3$ content and the water holding capacity (WHC) of the acidic sandy soil from Eastern Hungary at a low application rate. (IV, V)
    a. Bauxite residue mixed into the acidic sandy soil (ASS) at low doses (5–10 $^{w}/_{w}$ %) did not exceed the Hungarian quality criteria for soil based on KvVM-EüM-FVM Joint Decree No. 6/2009. (IV)
    b. 5–20 $^{w}/_{w}$ % bauxite residue increased the microbial activity of the acidic sandy soil over the short-term, but the effect did not last for long-term according to Biolog EcoPlate parameters [average well colour development (AWCD), substrate average well colour development (SAWCD), substrate richness (SR)]. Shannon diversity index (H) showed that bauxite residue at up to 20 $^{w}/_{w}$ % did not change microbial diversity over the short term, but the diversity decreased on the long-term. (IV, V)
    c. 5 $^{w}/_{w}$ % bauxite residue had no significant adverse effects on the tested test organisms [*Aliivibrio fischeri* (luminescent bacterium), *Sinapis alba* (plant), *Triticum aestivum* (plant) and *Tetrahymena pyriformis* (protozoon)]. (IV)

3. I established that the bauxite residue and soil mixture (BRSM) removed after the bauxite residue spill in Ajka (Hungary) and disposed in the dams at MAL Co. Ltd. in Ajka is applicable as additive to the surface layer of landfill cover systems at landfill sites at up to 20 $^{w}/_{w}$ % BRSM application rate in low quality subsoil, improving soil physico-chemical and biological characteristics and thus the settlement and growth of plants. (V)
    a. 5–20 $^{w}/_{w}$ % BRSM in the low quality subsoil had a long lasting enhancing effect on the microbial community based on all Biolog EcoPlate parameters. (V)
    b. The ecotoxicity test results of leachate and soil showed that 20 $^{w}/_{w}$ % BRSM had no adverse effect on the following test organisms: *Sinapis alba*, *Triticum aestivum*, *Aliivibrio fischeri* and *Folsomia candida* during the experiments. (V)

4. I demonstrated that the Biolog Ecoplate derived evaluation parameters gave a reliable response to both the positive and negative effects of the bauxite residue on the soil microflora; moreover, these results extendable to the microbial activity and diversity of the soil microflora according to literature.

    To assess the positive and negative effects of similar soil amendments on the microflora of deteriorated soils the calculation of the AWCD (average well colour development) and SAWCD well colour development), H (Shannon-index) and SR (substrate richness) values is recommended. (III)

Evaluation the bauxite residue as a source of critical raw materials:

1. I developed a state-of-the-art-technology for extraction of critical raw materials (CRM) including rare earth element (REE) from Hungarian bauxite residue. (I)
    a. Optimal extraction parameters maximizing the economic potential (total metal extracted × economic value of the respective metal) of CRM were determined using a design of experiment approach (5.6 M HCl, 24 h, 73.4 °C, 100 g $L^{-1)}$. (I)

    b. These conditions allowed to extract an overall economic potential of 40.95 ± 0.90 US \$ t^{-1}, corresponding to 64% of the total economic potential of CRM present in Hungarian bauxite residue. (I)

2. I proved that ultimately more than > 40% of the overall rare earth elements (REEs) (> 62% of the leachable REEs) in Hungarian bauxite residue could be purified using liquid-liquid extraction, whereas Al was successfully rejected from the concentrate (~5% of the overall Al present) by precipitation. (II)
    a. Optimal extraction conditions maximizing the economic potential (total metal extracted × economic value of the respective metal) of CRM were determined using a design of experiment approach (0.1 for LLE organic/aqueous ratio, 0.78 M for D2EHPA concentration in kerosene, 0.67 for stripping organic/aqueous ratio and 4 M for HCl concentration). (II)
    b. The prediction model was accurate, since the experimentally determined economic potential corresponded (88%) with the predictions and allowed a maximum recovery of 17.18 ± 0.59 US \$ t^{-1}. (II)

6. Conclusions, potential applications

Bauxite residue is the major by-product produced during alumina production following the Bayer's process. The treatment and disposal of this residue is a major operation in an alumina plant. A lot of research and developmental activities are going on throughout the world to find effective utilization of bauxite residue, which involves various product developments.

Because of the bauxite residue disposal in large quantities also in Hungary, this research assessed and evaluated the efficiency of a Hungarian bauxite residue applied as soil ameliorant on the one hand, and as a secondary source of CRM on the other hand.

The combined assessment demonstrated that the 5 $^{w}/_{w}$ % bauxite residue mixed into the acidic sandy soil (ASS) was the most favourable treatment moreover both the analytical, biological and the toxicity measurements applied in the field study showed that up to 20 $^{w}/_{w}$ % BRSM mixed into the LQS had no adverse effects on the soil ecosystem.

The integrated methodology, consisting of physical, chemical, biological and ecotoxicological methods supported the efficient monitoring of technology performances and the identification of the advantages and limitations of bauxite residue and BRSM application for soil improvement.

In the next section of this research, a selective recovery method of CRM from Hungarian bauxite residue was developed. Several parameters for instance leaching agents, contact time, temperature, solid to liquid ratio, complexing agent concentration in organic phase were investigated to optimize the leaching and LLE.

The developed optimal conditions allowed extraction of the economic potential of 17.18 US \$ t^{-1}, corresponding to 27% of the total economic potential of CRM present in bauxite residue. It can be anticipated that exploitation of bauxite residues as secondary source for CRM will increase in the near future. Therefore, the methodology presented here is generally applicable also for other bauxite residues with different elemental composition, allowing to rapidly maximizing extraction efficiencies, considering also minor and / or interaction effects of extraction parameters also in further bauxite residues. Considering the sum of potential economic values extracted (rather than mere extraction efficiencies) directly allows reacting to volatility in different commodity prices.

7. Publications

Journal articles on which the thesis was based

I. **Ujaczki É**., Zimmermann Y.S., Gasser C.A., Molnár M., Feigl V. & Lenz M. (2017) Red mud as secondary source for critical raw materials – Extraction study. J. Chem. Tech. Biotech. DOI: 10.1002/jctb.5300. (IF:3.14; I:1)

II. **Ujaczki É**., Zimmermann Y.S., Gasser C.A., Molnár M., Feigl V. & Lenz M. (2017) Red mud as secondary source for critical raw materials – Purification of rare earth elements by liquid/liquid extraction. J. Chem. Tech. Biotech. DOI: 10.1002/jctb.5289. (IF:3.14; I:1)

III. Feigl V., **Ujaczki É**., Vaszita E. & Molnár M. (2017) Influence of red mud on soil microbial communities: Application and comprehensive evaluation of the Biolog EcoPlate approach as a tool in soil microbiological studies. Sci. Total Environ. 19 (595), 903–911. DOI: 10.1016/j.scitotenv.2017.03.266. (IF:5.10; I:1)

IV. **Ujaczki É**., Feigl V., Farkas É., Vaszita E., Gruiz K. & Molnár M. (2016) Red mud as acidic sandy soil ameliorant: a microcosm incubation study. J. Chem. Technol. Biotechnol. 91, 1596–1606. DOI: 10.1002/jctb.4898. (IF:3.14; I:5)

V. **Ujaczki É**., Feigl V., Molnár M., Vaszita E., Uzinger N., Erdélyi A. & Gruiz K. (2016) The potential application of red mud and soil mixture as additive to the surface layer of a landfill cover system. J. Environ. Sci. 44, 189–196. DOI: 10.1016/j.jes.2015.12.014. (IF:2.94; I:6)

VI. **Ujaczki É**., Klebercz O., Feigl V., Molnár M., Magyar Á., Uzinger N. & Gruiz K. (2015) Environmental toxicity assessment of the spilled Ajka red mud in soil microcosms for its potential utilisation as soil ameliorant. Period. Polytech. Chem. 59 (4), 253–261. DOI: 10.3311/PPch.7839. (IF:0.55; I:9)

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Feigl V., Molnár M., **Ujaczki É**., Klebercz O., Tolner M., Vaszita E. & Gruiz K. (2015) Ecotoxicity of biochars from organic wastes focusing on their use as soil ameliorant. AquaConSoil 2015 Conference Proceedings, 13th International UFZ-Deltares Conference on Sustainable Use and Management of Soil, Sediment and Water Resources, June 9–12, 2015, Copenhagen, Denmark.

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Molnár M., Vaszita E., Farkas É., **Ujaczki É**., Fekete-Kertész I., Tolner M., Klebercz O., Kirchkeszner Cs., Gruiz K., Uzinger N. & Feigl V. (2016) Acidic sandy soil improvement with biochar – A microcosm study. Sci. Total Environ. 563–564, 855–865. DOI: 10.1016/j.scitotenv.2016.01.091. (IF:5.10; I:11)

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