



MME SEE

CONGRESS 2023

5th Metallurgical & Materials Engineering
Congress of South-East Europe
Trebinje, Bosnia and Herzegovina
7-10th June 2023

CONGRESS PROCEEDINGS

MME SEE

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**5th Metallurgical & Materials Engineering
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PROCEEDINGS**

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PREFACE

On behalf of the Scientific and Organizing Committee, it is a great honor and pleasure to wish all the participants a warm welcome to the Fifth Metallurgical & Materials Engineering Congress of South-East Europe (MME SEE 2023) which is being held in Trebinje, Bosnia and Hercegovina, 07 - 10 June 2023.

The MME SEE 2023 is a biannual meeting of scientists, professionals, and specialists working in the fields of metallurgical and materials engineering. The aim of the Congress is to present current research results related to processing/structure/property relationships, advances in processing, characterization, and applications of modern materials. Congress encompasses a wide range of related topics and presents the current views from both academia and industry: Future of metals/materials industry in South-East European countries; Raw materials; New industrial achievements, developments and trends in metals/materials; Ferrous and nonferrous metals production; Metal forming, casting, refractories and powder metallurgy; New and advanced ceramics, polymers, and composites; Characterization and structure of materials; Recycling and waste minimization; Corrosion, coating, and protection of materials; Process control and modeling; Nanotechnology; Sustainable development; Welding; Environmental protection; Education; Accreditation & certification.

The editors hope that Congress will stimulate new ideas and improve knowledge in the field of metallurgical and materials engineering. The Congress has been organized by the Association of Metallurgical Engineers of Serbia, with the co-organization of the Institute for Technology of Nuclear and Other Mineral Raw Materials, Belgrade, Serbia, Faculty of Technology and Metallurgy, University of Belgrade, Serbia, Faculty of Technology, University of Banja Luka, Bosnia and Herzegovina; the Faculty of Metallurgy, University of Zagreb, Sisak, Croatia; the Faculty of Natural Sciences and Engineering, University of Ljubljana, Slovenia; and the Faculty of Metallurgy and technology, University of Podgorica, Montenegro.

Financial support from the Ministry of Science, Technological Development and Innovation of the Republic of Serbia to researchers from Serbia for attending the congress is gratefully acknowledged. The support of the sponsors and their willingness to cooperate have been of great importance for the success of MME SEE 2023. The Organizing Committee would like to extend their appreciation and gratitude to all sponsors and friends of the conference for their donations and support.

We would like to thank all the authors who have contributed to this book of abstracts and also the members of the scientific and organizing committees, reviewers, speakers, chairpersons, and all the conference participants for their support of MME SEE 2023. Sincere thanks to all the people who have contributed to the successful organization of MME SEE 2023.

On behalf of the 5th MME SEE Scientific and Organizing Committee

Miroslav Sokić, PhD

REE EXTRACTION FROM HYPERACCUMULATING PLANTS: CHALLENGES AND PROSPECTS

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Phytomining although predominantly in its early stages on the broader scientific scope of investigation has garnered interest in metals such as Ni, Au, Zn or REE. Rare earth elements (REE) are commonly defined as the 15 lanthanides with the addition of Sc and Y. Since studies of other hyperaccumulating plant species have been conducted before, similar techniques were tried with REE. With the concentration threshold at 0.1 wt% of the dry biomass there have been identified about 22 plant species that hyperaccumulate REE from five families (Phytolaccaceae, Gleicheniaceae, Blechnaceae, Juglandaceae, Thelypteridaceae). One of the most studied REE hyperaccumulators is *Dicranopteris linearis* (formerly known as *D.dichotoma*), a fern native to China that grows on mine tailings. Generally, the techniques for REE phytomining can be separated into Pyrometallurgy, Hydrometallurgy, Biometallurgy and Novel approaches. Currently most of the research is still focusing on the optimal utilization of various hiperaccumulators and the enchantment of REE extraction from the soil as well as from the enriched biomass. Since recovery of REEs via phytomining is just in its infancy further multidisciplinary investigations are needed, especially when it comes to valorization and REE recovery. This paper presents a quick review for the concept of REE phytomining, current state of research, challenges and prospects.

Keywords: Phytoextraction, Rare earth metals, Secondary resources

Introduction

Rare earth elements (REEs), are chemical elements consisting of 15 lanthanides as well as yttrium and scandium, share similar chemical properties. Their unique characteristics and wide range of application in modern technologies have resulted in substantial growth in production over the past two decades [1]. REEs are divided into two groups due to the differences in atomic mass: light rare earth elements (LREEs) and heavy rare earth elements (HREEs). LREEs are La, Ce, Pr, Nd, Sm, Eu, and Sc, while HREEs are Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, and Y [2].

REE raw materials are usually obtained from such types of ores such as: monazite (China, USA, Australia, India, Malaysia, Brazil, Thailand and Sri Lanka), bastnaesite (USA, China) and laterite ores, from which REE concentrates can be obtained by means of flotation, magnetic and gravity enrichment [3]. In terms of naturally occurring elements like silver or gold, REE constitute the seventh most unreplenishable resource. They are not as rare as their name suggests, REE are widely found in the Earth's crust with Cerium being the most abundant one. The name refers rather to the fact that the REE deposits are very scattered and it is difficult to isolate them.

REEs occur in nature in their oxidized form in minerals and salts because of their electropositive nature and high affinity for oxygen. Globally the supply of REEs is dominated by China, who is the source of up to 97% of rare earth production [4]. The increased use of REE also leads to an increase in the amount of these elements in the environment. Exploitation of REE causes their increased migration in the environment, and this leads to increased volume of tailings or mining waste [5, 6]. Incentives from the Chinese government for the reclamation of these tailings benefitted the research on the phytomining of REEs. These tailings are the most studied substrate for wild natural growth of REEs hyperaccumulators on a medium scale.

Over four hundred plant species classified as natural metal hyperaccumulators have been identified, but the list of species is still being developed [3]. When it comes to REE an accumulation limit of 100 mg kg⁻¹ dry weight was established for the total sum of REE present in the plant biomass [7], and over twenty species have been identified as REE hyperaccumulators.

Some examples of REE hyperaccumulating plants include *Phytolacca americana* (Phytolaccaceae), *Dicranopteris linearis* (Gleicheniaceae), *Woodwardia japonica* (Blechnaceae), *Carya cathayensis* (Juglandaceae), *Pronephrium triphyllum* (Thelypteridaceae) [8]. One of them, *Dicranopteris linearis* (syn. *Dicranopteris dichotoma*) has been the focus of most studies as it has been recorded naturally colonizing REE tailings in China.

The aim of this paper is to provide concise review for the current state of REE phytomining technology, its perspectives, challenges and future prospects. Special attention is paid to the developing technologies for REE extraction from hyperaccumulating plant biomass.

Results and discussion

Phytomining of REEs

REE polluted soils are found with REE mines, coal mines, agricultural soils treated with phosphate fertilizers, and roadsides. It has been noted that REEs tend to concentrate in upper part of the soil horizon, making them suitable for the phytorecovery process [5]. Only certain part of the REE pool in soil is bioavailable to plant organisms. In terms of binding with organic matter, REE behave in soils similarly to other trace elements. Cation exchange capacity, organic matter content, pH, and metal content in the substrate are some of the soil factors that can affect hyperaccumulation. Soil pH has a significant impact on the growth and development of hyperaccumulating plants. Hyperaccumulating plants are found by examining bioaccumulation and translocation factors, soil to root and root to shoot metal ratio, respectively. While establishing an accumulation threshold to be crossed, both factors should be >1 for REE. REEs are not essential for plants, in low doses they can stimulate seed germination, plant growth and production of chlorophyll, while in high concentration they may negatively affect plant growth. As a result, it may not be possible to recover satisfactory amount of REEs along with the biomass.

In order to make REE phytomining a feasible process, few requirements should be met. The first one is to use this technology on sites naturally or anthropogenically enriched with REE. The second is to promote bioavailability to plants by adding assisting amendments (such as fertilizers, acids or chelates). The third part is selection of adequate plant species with ability to hyperaccumulate selected REEs, that should be native to the exploitation area. In order to gain sufficient amount of biomass with accumulated elements, sometimes specific agronomic measures should be taken [9]. Certain researched show that, for reaching the economic viability, biomass production needs to reach over 10 t/ha [10]. Upon the harvesting aboveground biomass, further steps are connected to the extraction technologies.

A perfect example of naturally resistant potent species for the phytoextraction of REE is *Phytolacca americana* that occurs naturally in the United States and can reach up to 3 meters in height [11]. The bioaccumulation factor (BF) and translocation factor (TF) of *Phytolacca americana* were higher than one, and the REE accumulation follow the decreasing order of leaf $>$ root $>$ stem. According to a recent field survey *Phytolacca americana* (pokeweed) is indicated to achieve 1040 mg REE per kg 1 of leaves [12]. Some other pteridophytes that have satisfactory BF >1 for REE are *Asplenium filipes*, *A. trichomanes*, *Blechnum subnormale*, *Driopteris fucipes* etc. [13].

Extraction of rare earth elements

Implemented extraction technologies for REE from phytomining are currently pyrometallurgy, hydrometallurgy, biometallurgy and novel approaches, and their overview is presented in Fig 1. However, applying one single approach is usually not sufficient for complete separation and purification of REE.

Pyrometallurgy is a well-known thermal method that has been in practice for years because of its wide-range applications, simplicity, and huge capacity, but it implies high energy consumption and has raised additional environmental challenges. In phytomining technology the thermal conversion of contaminated biomass can be divided into pyrolysis (350–700 °C with the absence of oxygen), ashing (300–550 °C), combustion (over 900 °C with excess oxygen/air), and gasification (700–1000 °C with partial oxidation) [14]. Hydrometallurgy uses a chemical method to recover metals by means of dissolution and leaching that are followed by the separation and purification of said metals. Leaching is a complex process that can be affected by a multitude of factors such as temperature, time, solid-to-liquid ratio, pH, lixiviant type, particle size, agitation speed, while purification is mostly limited by the removal of undesired metals present in the leaching solution. Bioleaching refers to usage of bacteria, microorganisms, fungi, algae, or their metabolites to interact with metals, and is usually used as a substitute in certain part of separation processes in both hydrometallurgy and pyrometallurgy [15]. Novel approaches refer to research that has been even partially successful in metal extraction from other secondary resources but have not yet been tested on biomass from phytomining, such as ionic liquid, electrochemical, mechanochemical, and supercritical fluid extractions.

	Pyrometallurgy	Hydrometallurgy	Biometallurgy	Novel approaches
Factor	- Pyrolysis (350–700 °C no oxygen)	- Leaching (temperature, time, solid-to-liquid ratio, pH, lixiviant type, particle size, agitation speed)	- Bacteria	- Ionic liquid
	- Ashing (300–550 °C)	- Purification (Selective precipitation, filtration or other separation techniques)	- Microorganisms	- Electrochemical
	- Combustion (> 900 °C excess oxygen/air)		- Fungi	- Mechanochemical
	- Gasification (700–1000 °C partial oxi)		- Algae	- Supercritical fluid extractions

Figure 1 Potential extraction technologies of REE from contaminated plants

When it comes to extraction of REE there have been several challenges when compared to previous experiments of phytomining of metals such as Ni. Unfortunately to this day the only REE hyperaccumulator that has undergone published extraction studies is *D. linearis*.

Due to high contents of Si and Al in *D. linearis* its ashing results in an amorphous aluminosilicate matrix. This matrix stabilized REEs and that made effective acidic leaching nearly impossible [16]. This problem has been treated in two ways: one was to recover REEs directly from the dry biomass [17], and the other was to make REE available for extraction after ashing by removing aluminum and exposing the encapsulated metals [18]. The approaches to REE extraction from *D. linearis* has been illustrated in Fig. 2. Since Si hinders leaching after ashing and Al requires corresponding separation making REE leachate purification difficult, other hyperaccumulating species could pose more acceptable material for the REE extraction. For instance, **P. americana** presents a strong candidate for future REE extraction research, as it has much less Si (780 µg/g) than *D. linearis* (14,000 µg/g) that could enhance the leaching process [19].

The first research used ion exchange resin for the purification step after leaching process of REE from *D. linearis* biomass [20]. Authors used the dried and crushed biomass of the *D. linearis* collected from REE mine tailings in China. The biomass was initially leached in 0.5 M nitric acid solution with the presence of exchange resin, and the REE were absorbed into the resin. This was followed by washing the resin with water and 0.75 M HNO₃.

At the end the REE were removed from the resin by using 3 M HNO₃ yielding a solution containing 81.4% REEs purity. Their work was a basis for the research of Laubie et al. [16] that explored a hydrometallurgical pathway to recycle REEs from *D. linearis* growing naturally on former mine tailings by directly using an EDTA solution (ethylenediaminetetraacetic acid) on the dried biomass and precipitating REEs from the leaching solution using oxalic acid. They found that optimal conditions that resulted in efficiency of 85% are that leaching be performed with 0.05 M EDTA solutions, at a solid-liquid ratio (S/L) of 30 g/L, for a duration of 2 h, while precipitation be conducted at pH 2.3 with an oxalic acid molar ratio of 8:0.37.

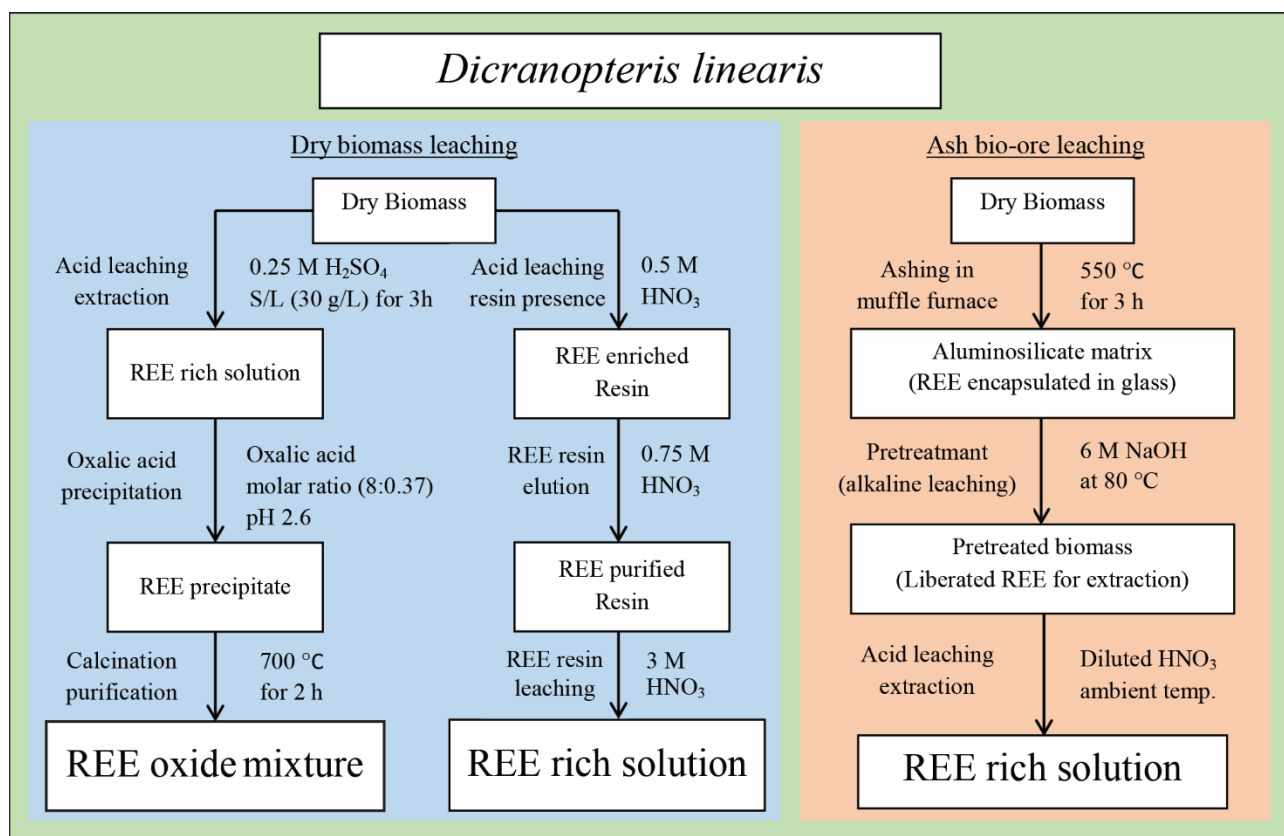


Figure 2. Recovery of REEs from *Dicranopteris linearis* biomass

This approach has been further developed by Chour et al. [17] in a comprehensive hydrometallurgical pathway for reclaiming REEs from the *D. linearis*. The collected dry fern biomass was leached with six aqueous solutions under the same conditions of S/L = 30 g/L, and 2 hours of magnetic stirring. The used solutions were deionized water, sulfuric acid, citric acid, citrate, generic EDTA, and modified EDTA. The most effective leaching solution was sulfuric acid at a concentration of 0.25 M H₂SO₄. The precipitation with oxalic acid after leaching had the same molar ratio as the previous study Laubie et al. [16] but the pH adjustment was slightly higher at 2.6. The precipitate was calcinated at 700 °C for 2h to convert it into a mixture of rare earth oxides as the final product. This newly developed hydrometallurgical pathway resulted in a recovery of 72% of REEs from the plant. As mentioned before the key challenge for an efficient extraction of REE from *D. linearis* ash is an efficient and early removal of Al, by dissolving the aluminosilicate matrix akin to the Bayer process. This was the inspiration for Jally et al. [18] who proposed a three-step extraction process: hot caustic soda solution is used to dissolve the aluminosilicate matrix of the ash and increase REE availability, then the remaining solid is rinsed with water to lower the pH, and finally the rinsed solid is leached using nitric acid (HNO₃) to extract the REEs.

The harvested *D. linearis* was initially dried at 80 °C for 72 h before ashing at 550 °C for 3 h in a muffle furnace to secure complete oxidation. The collected ashes were subjected to pre-treatment and dissolution. Pretreatment consisted of using 6 M sodium hydroxide (NaOH) at an average temperature of 80 °C, after which the pretreated biomass was washed with water in order to prepare for dissolution. REEs were extracted by employing diluted nitric acid (HNO₃) at ambient temperature. There were several losses of REEs during the leaching and purification steps. The first (around 12%) occurred prior to acid leaching, but could not be contributed to any specific step of factor. The second loss was noticed in the HNO₃ leaching step (around 30%) as a result of the compromise between REE recovery and keeping Al in the solid phase. Nevertheless, the recovery rate of the extraction procedure was reported at 74% under optimal conditions, but at present NaOH costs in the alkaline leaching step make the whole process unprofitable at laboratory scale.

Conclusion

The importance of REE for our everyday life is multifaceted as they are used across various industries. The largest consumer of REE raw materials is the glass industry that uses cerium oxide for polishing and many REE as additives that provide color (yttrium, europium, and terbium for red-green-blue) and special optical properties (lanthanum in digital cameras). Neodymium-iron-boron magnets are the strongest magnets developed for commercial computing industry, but are also used in automotive subsystems (power steering, electric windows, power seats, and speakers). Mischmetal used to remove impurities during steel production of special alloys is mixture of REE oxides (cerium, lanthanum, neodymium, and praseodymium).

Alternative methods of secondary resource exploitation are on the rise, phytomining being one of them with the special focus on the green agenda. About 22 plant species that hyperaccumulate REE have been identified from *Phytolaccaceae*, *Gleicheniaceae*, *Blechnaceae*, *Juglandaceae*, *Thelypteridaceae* families. Since the only extraction research of REE from plant biomass has been performed on a fern species *Dicranopteris linearis* it would be beneficial to see how those hydrometallurgical pathways would prove on other potent REE hyperaccumulators such as a pokeweed species *Phytolacca Americana*. Regardless of the extraction method employed the REE yields from *D. linearis* are between 70 and 80% with costly resource consumption. Unfortunately, the loss of extraction yield can't be pinpointed to one specific step, which is usual for early development research.

Unlike Ni phytoextraction which is in semi-industrial research, REE research is still focused on a few plant species capable of accumulating REE and making their biomass satisfactory for an economically viable process. Furthermore the couple of extraction pathways that have been studied on REE hyperaccumulators have significant setbacks in the areas of separation, purification, and production and this too poses challenges to the economic viability of REE recovery from secondary sources. It is clear that more study is needed on the optimization of REE phytoextraction, however, it is certainly a prospective field for future research.

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1. Association of Metallurgical Engineers of Serbia (Beograd)

- a) Металургија – Зборници
- b) Технички материјали – Зборници
- c) Наука о материјалима – Зборници
- d) Металопрерађивачка индустрија – Зборници

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