

# Extraction of ammonium nickel sulfate hexahydrate by hydrometallurgical process from the hyperaccumulating plant *Odontarrhena muralis* – case study from Serbia

Branislav Marković<sup>1</sup>, Dragana Ranđelović<sup>1</sup>, Gvozden Jovanović<sup>1</sup>, Gordana Tomović<sup>2</sup>, Ksenija Jakovljević<sup>2</sup>, Tomica Mišljenović<sup>2</sup> and Miroslav Sokić<sup>1</sup>

<sup>1</sup>Institute for Technology of Nuclear and Other Mineral Raw Materials, Franchet d'Esperey Boulevard 86, Belgrade, Serbia

<sup>2</sup>Institute of Botany and Botanical Garden, Faculty of Biology, University of Belgrade, Takovska 43, Belgrade, Serbia

## Abstract

Phytomining is a new promising technique that is based on using hyperaccumulating plants which biomass is utilized as a bio-ore for metal extraction. The Ni-hyperaccumulating species *Odontarrhena muralis* is widely distributed on ultramafic soils in Serbia, and could be a promising candidate for Ni agromining. In the present study, efficiency of a hydrometallurgical process for Ni recovery using biomass of *O. muralis* wild population through the synthesis of Ni salts from plant ash in the form of ammonium nickel sulfate hexahydrate,  $\text{Ni}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$  – (ANSH) was assessed. The average Ni content in the plant from ultramafic sites in West Serbia was up to  $3.300 \text{ g kg}^{-1}$ . The mass yield of ANSH crystals from the crude ash was ~12 % with the average purity of 73 % were obtained. By optimizing the purification process before precipitation of ANSH crystals, it is possible to obtain salt crystals of higher purity, which increases the economic profitability of this process. The results of this preliminary study on wild population of *O. muralis* show the increased potential for implementation of phytomining practices as an alternative way of Ni extraction on ultramafic sites in Serbia.

**keywords:** phytomining, bio-ore, leaching, nickel extraction.

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## 1. INTRODUCTION

One of the main consequences of development and increasingly intensive anthropogenic activities is the generation of vast quantities of waste, often containing significant concentrations of potentially toxic elements (*e.g.* heavy metals). At the same time, intensive mining significantly depletes existing ore reserves, making the ores more expensive to extract and process. Therefore, in recent decades, new techniques have been developed that allow recycling of metal(oid)s, *i.e.* extraction from waste materials or from substrates with concentrations of metals or their compounds not sufficient for economically acceptable mining. One such technique is phytomining, in which selected hyperaccumulating plants are grown on a metal-bearing substrate with the aim of concentrating high amounts of the metal(oid)s in the biomass [1]. After the biomass is harvested, it is used as a bio-ore for metal(oid)s extraction. This process is similar to phytoextraction, with the difference that the biomass obtained by classical phytoextraction is treated as waste, while in the process of phytomining, it is a raw material for the recovery of metals or their compounds [2]. Hyperaccumulator plant species are those capable of accumulating metal(oid)s in the aboveground tissues at concentrations 100 – 1000-fold higher than in “normal” plants and higher than the threshold specifically proposed for most potentially toxic elements [3]. Hyperaccumulation of metal(oid)s is a rather unusual and rare phenomenon that has been described in about 700 vascular plants [4]. Most of these species hyperaccumulate Ni, almost exclusively on ultramafic soils, which are uninhabitable for many plant species due to the soil specific physical and chemical properties. The largest ultramafic areas in Europe are

Corresponding author: Branislav Marković, Institute for Technology of Nuclear and Other Mineral Raw Materials, Franchet d'Esperey Boulevard 86, Belgrade, Serbia

E-mail: [b.markovic@itnms.ac.rs](mailto:b.markovic@itnms.ac.rs)

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located in the Balkans, as large blocks or smaller outcrops [5] with significant representation in West and South-West Serbia. To be considered a hyperaccumulator of Ni, a plant has to contain more than  $1.00 \text{ g kg}^{-1}$  Ni in the dry matter of the aboveground tissues when growing in its natural habitat [3]. The largest number of Ni hyperaccumulators has been found in the genus *Odontarrhena* C. A. Mey ex Ledeb. (syn. *Alyssum* L. pro parte, sect. *Odontarrhena* (Meyer) Koch). One hyperaccumulator plant species belonging to this genus is *Odontarrhena muralis* (Waldst. & Kit.) Endl. (syn. *Alyssum murale* L.), which has been shown to accumulate up to  $20.000 \text{ g Ni per kg dry weight}$  [1]. Nickel concentrations of up to  $2.926 \text{ g kg}^{-1}$  in roots,  $6.793 \text{ g kg}^{-1}$  in shoots and  $13.160 \text{ g kg}^{-1}$  in leaves of *O. muralis* were found at different ultramafic sites in Serbia [6].

Ash from the plant combustion can be regarded as a bio-ore from which Ni or its products can be obtained either by pyrometallurgical or hydrometallurgical means. Li and co-workers have explored the pyrometallurgical extraction of metallic Ni by feeding biomass or ash directly into a smelter [7], while the hydrometallurgical extraction of metallic Ni can be performed from a leachate solution [8,9]. Not only metallic Ni, but also its various compounds can be recovered from plant ash, such as Lewis acid catalysts [10,11] or various Ni salts (ammonium nickel sulfate hexahydrate [12], nickel sulfide [13] or nickel hydroxide [14]). However, precipitation of Ni by direct leaching using water proved to be ineffective due to complexation of Ni with stronger ligands [15]. Ultimately, biomass ashing has shown promising results provoking various research studies related to optimization the ashing temperature, leaching agents, stirring rate, acid concentration, and other operating parameters towards more economically feasible and environmentally sustainable solutions [16,17].

After effective leaching, once an aqueous solution of  $\text{NiSO}_4$  is obtained in sufficient concentration and purity, Ni products can be obtained in numerous forms, starting with  $\text{NiSO}_4$  itself. With the addition of ammonium sulfate  $(\text{NH}_4)_2\text{SO}_4$  to the solution, ammonium nickel sulfate hexahydrate can be obtained, or nickel hydroxide  $\text{Ni}(\text{OH})_2$  can be precipitated by a suitable base such as  $\text{MgO}$ ,  $\text{NaOH}$  or  $\text{K}_2\text{CO}_3$ . In addition, nickel hydroxide can be used as a final product or converted into nickel sulfamate  $(\text{Ni}(\text{NH}_2\text{SO}_3)_2)$ , which is used for nickel electroplating, or it can be converted into nickel citrate  $(\text{Ni}_3(\text{C}_6\text{H}_5\text{O}_7)_2)$  using citric acid  $(\text{C}_6\text{H}_8\text{O}_7)$ . Taking into account that all these Ni products must be of the highest purity (above 98 %) to be considered commercially viable, ammonium nickel sulfate hexahydrate,  $\text{Ni}(\text{NH}_4)_2(\text{SO}_4)_2 \times 6\text{H}_2\text{O}$  – (ANSH) is a suitable Ni electroplating salt that can be obtained as a hydrometallurgical upgrade of Ni resulting from the phytomining process [18,19]. As grades of global Ni ore reserves continue to decline, the last decade has seen a shift from high-grade, low-volume ores to low-grade, high-volume ores. This decline has led to even greater volumes of waste produced by the mining industry, creating larger areas in need of remediation [20]. Bio-ores such as plant ash have a higher Ni concentration than common lateritic ores and do not contain Fe and Mg silicates, which are present in the soil matrix and could increase the cost of metal extraction. This fact shows a double benefit of phytomining; not only it helps in the remediation of waste contaminated soil, but it also produces higher-grade Ni bio-ores [21]. Unfortunately, despite the scientific validation of phytomining, the mining industry has not conducted testing beyond the pilot scale required to prove viability of this process in "real life". New research trends in the field of phytomining are moving toward more specifically designated studies to field conditions and valorization of bio-ores in terms of profit and environmental safety [22,23].

Several long-term studies have been conducted to optimize agronomic techniques for *Odontarrhena* species cultivation and Ni extraction from the low-productive ultramafic soils [24–26]. Mineral fertilizers (N, P, K) were found to be the most efficient, although significant results were also obtained with co-cropping and manure application [27–29]. Large variability of Ni concentrations in plant organs of *Odontarrhena* spp. clearly indicates that substrate properties (pH, amount of organic matter, water capacity, etc.) are important factors for development of these species [30]. Genetic structure of local populations also affects greatly the amount of Ni extracted from the soil. In view of this, it is of particular importance to conduct experiments at the local level to assess the potential for efficient metal recovery from native populations. Development of a customized process of *O. muralis* cultivation, biomass collection, and Ni extraction could lead to a sustainable and environmentally friendly methodology for Ni extraction from low-productive ultramafic outcrops in Serbia. This study aimed to test the efficiency of a hydrometallurgical process for Ni recovery from wild population of *Odontarrhena muralis* biomass through synthesis of Ni salts in the form of ammonium nickel sulfate hexahydrate,  $\text{Ni}(\text{NH}_4)_2(\text{SO}_4)_2 \times 6\text{H}_2\text{O}$  – (ANSH) from the plant ash. This study represents a preliminary attempt to develop a method for Ni phytoextraction from hyperaccumulating plant populations in Serbia.

## 2. EXPERIMENTAL

Aboveground biomass of *O. muralis* was collected during the flowering phase in July 2019 at an ultramafic site in West Serbia (Mt Maljen, Tometino polje). The plant material was separated into leaves (OML), stems (OMS), and flowers (OMF), as well as the rest of the whole plant biomass and air-dried. The elemental composition of the plant material, ash, effluents, and leachates was analyzed by atomic absorption spectrometry (PinAAcle 900T PerkinElmer, Inc, Norwalk, CT, USA) with three replicates. Chemical elements of interest in this study were: Ni (as the only valorizing element), K, Ca (elements present in the plant that may interfere with the Ni extraction), Fe, and Mg (elements that may compete with Ni and reduce the purity of the final products). To determine contents of these elements in the plant biomass, the samples were priorly dissolved using the nitric acid-perchloric acid digestion method [31], and the inorganic elements (Ni, Ca, Mg, Fe, and K) were determined directly from the resulting solution. However, for determination of the Ni content in ash samples, the acid digestion method using aqua regia was applied [32], while for the remaining elements (Ca, Mg, Fe, and K), an alkali digesting method using HF+HClO<sub>4</sub> followed by H<sub>2</sub>O+HCl dissolution was used [33]. For liquid samples such as effluents and leachates, the elemental content was determined directly by atomic absorption spectrometry. Standard Carl Roth solutions were used to calibrate the absorption curves for all sample types and elements. Apple leaf standard reference material NIST 1515 was used to verify the accuracy of the method.

Following the procedure developed by Zhang *et al.* [13] for more efficient extraction of Ni, the air-dried biomass was burned at 550 °C for 4 h. Potassium was removed from the ash by washing, which was repeated once with deionized water at a solid-liquid ratio of 1:4 for a period of 15 min using hand agitation. After washing, leaching of Ni from the ash was carried out using 2 M sulfuric acid (96 % *p.a.* Lachner, Czech Republic) heated to 95 °C with a solid-liquid mass ratio of ash to the acid of 1:9. Before crystallization of ANSH from the leachate, a two-step purification was performed to remove Fe and Mg. In the first step, Ca(OH)<sub>2</sub> (*p.a.* Sigma-Aldrich Chemie GmbH, Germany) was added at the concentration of 10 % to the leachate for neutralization and Fe removal. In the second step, NaF (*p.a.* Sigma-Aldrich Chemie GmbH, Germany) was added in excess of 10 % to remove Mg from the leachate. After each purification step, the leachate was vacuum filtered. Finally, to crystallize ANSH from the purified leachate, (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> (*p.a.* Merck, Germany) was added in excess of 20 %, and the solution was heated to 60°C to dissolve all salts. To increase the cooling and to crystallize higher amounts of ANSH, the solution was stored overnight at 4 °C.

Analysis of ANSH crystals and the solution after crystallization was performed by inductively coupled plasma optical emission spectrometry ICP-OES (Spectro Genesis, USA), using Smart Analyzer Vision Software. All chemical analyses were performed using three replicates.

Mineral phase composition of the samples was determined by using X-ray diffraction (XRD). XRD patterns were obtained on an automated diffractometer (PHILIPS PW-1710, Eindhoven, The Netherlands) using a Cu tube operated at 40 kV and 30 mA. The instrument was equipped with a diffracted beam curved graphite monochromator and a Xe-filled proportional counter. Diffraction data were collected in the 2 θ Bragg angle range from 4 to 65°, counting for 1 s (qualitative identification) at every 0.02° step. Divergence and receiving slits were fixed at 1 and 0.1, respectively. All XRD measurements were performed at room temperature in a stationary sample holder.

Scanning electron microscopy (SEM) analyses were performed using a JEOL JSM-6610LV scanning electron microscope (JEOL Inc., USA) equipped with an INCA energy-dispersive X-ray analysis unit (EDS). An acceleration voltage of 20 kV was used. The analyzed samples were coated with a fine layer of gold dust to be visible under the microscope.

## 3. RESULTS AND DISCUSSION

### 3. 1. Chemical analyses of dry plant biomass and raw ash

Chemical compositions of the leaves (OML), stems (OMS), and flowers (OMF) of *O. murale* are shown in Table 1, as well as chemical compositions of the total aboveground plant biomass and the ash produced during combustion. According to the results, the highest Ni concentration was found in the leaves of *O. muralis*, which is in agreement with the results of other authors [34,35].



Table 1. Element contents in stems (OMS), leaves (OML), flowers (OMF), total aboveground plant biomass (OM) and raw ash (RA)

Sample	Content, g kg <sup>-1</sup>				
	Ca	Fe	K	Mg	Ni
OMS	5.968 ± 0.0231	n.d.*	11.104 ± 0.0304	1.612 ± 0.0147	1.003 ± 0.0112
OML	23.901 ± 0.00666	0.0987 ± 0.0038	11.326 ± 0.0119	4.410 ± 0.0103	4.790 ± 0.0040
OMF	1.710 ± 0.0116	0.102 ± 0.0036	17.995 ± 0.0142	4.020 ± 0.00351	2.980 ± 0.00557
OM	12.923 ± 0.0135	0.102 ± 0.0036	13.609 ± 0.0536	32.908 ± 0.0145	3.115 ± 0.0483
RA	159.075 ± 0.0512	2.092 ± 0.0258	163.034 ± 0.0331	35.926 ± 0.0234	30.120 ± 0.0429

\*n.d.- non determined below threshold of 0.0005

The initial aboveground plant biomass of ~610 g (OM) was reduced to 45 g of raw ash (RA) during combustion, which means that ~93 % of the plant mass was burned. As shown in Table 1, the Ni content in ash is about 10-fold higher than that in the plant dry matter, which is consistent with literature data [16].

XRD analysis of the aboveground plant biomass (OM), shown in Figure 1, revealed the presence of the following mineral phases: bunsenite (NiO), aranite (K<sub>2</sub>SO<sub>4</sub>), alcite (CaCO<sub>3</sub>), dolomite (CaMg(CO<sub>3</sub>)<sub>2</sub>), apatite (Ca<sub>5</sub>(PO<sub>4</sub>)<sub>3</sub>(OH,Cl,F), gorgeyite (K<sub>2</sub>Ca<sub>5</sub>(SO<sub>4</sub>)<sub>6</sub>H<sub>2</sub>O), and fairchilde (K<sub>2</sub>Ca(CO<sub>3</sub>)<sub>2</sub>). The predominant phases are aranite, calcite, and fairchilde, while bunsenite and apatite are present in lower amounts while gorgeyite is represented the least. Dolomite could be also present in the sample since its strongest diffraction peaks overlap with the peaks of the other present phases.

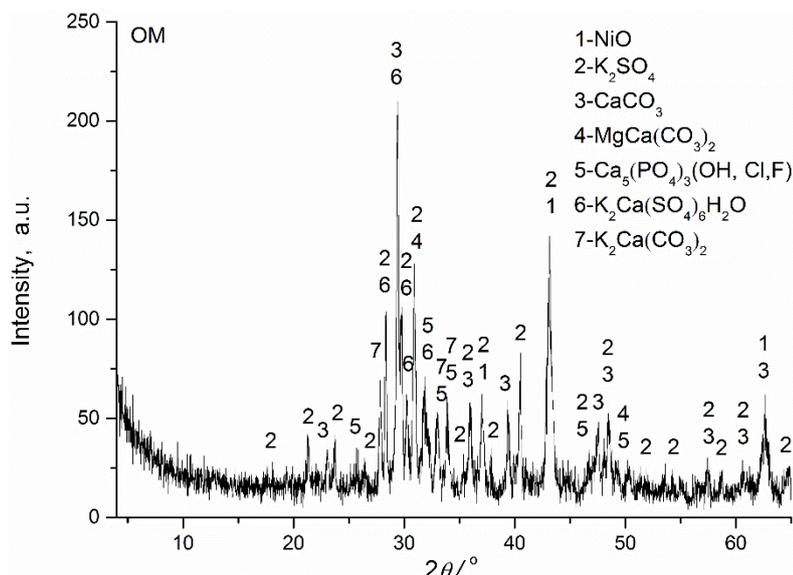


Figure 1. XRD analysis of the aboveground plant biomass (OM)

XRD analysis of raw ash (RA), presented in Figure 2, indicated the presence of the following mineral phases: calcite (CaCO<sub>3</sub>), bunsenite (NiO), arkanite (K<sub>2</sub>SO<sub>4</sub>), apatite (Ca<sub>5</sub>(PO<sub>4</sub>)<sub>3</sub>(OH,Cl,F), sylvite (KCl), K<sub>2</sub>CO<sub>3</sub>, fairchilde (K<sub>2</sub>Ca(CO<sub>3</sub>)<sub>2</sub>), panasqueiraite CaMgPO<sub>4</sub>(OH), and K<sub>2</sub>SO<sub>4</sub>CaSO<sub>4</sub>·5H<sub>2</sub>O. The predominant phase is calcite, while somewhat less present phases are bunsenite, aranite, K<sub>2</sub>CO<sub>3</sub>, and fairchilde.

As the XRD patterns show (Figs. 1 and 2), mineral compositions before (OM) and after combustion (RA) are not significantly different, the only difference being concentrations (Table 1). In addition, the SEM-EDS analysis of the RA sample (Fig. 3) confirmed the presence of nickel at the high concentration of 3.87 wt %, validating the results obtained by the chemical analysis. Morphology of the analyzed ash surface is similar to those found in other studies [16].

Effects of combustion on raw ash properties were reported in literature, moving from a lab muffle furnace to a pilot-scale boiler in scale-up studies [17]. Although ash quantity and quality were altered due to differences in temperatures and residence times, after washing and acid leaching, these differences did not affect Ni recovery in the leachate. While it was shown that using a pilot or industrial furnace would be efficient for burning large amounts of biomass, further research should be conducted to investigate potential energy recovery from the biomass combustion.

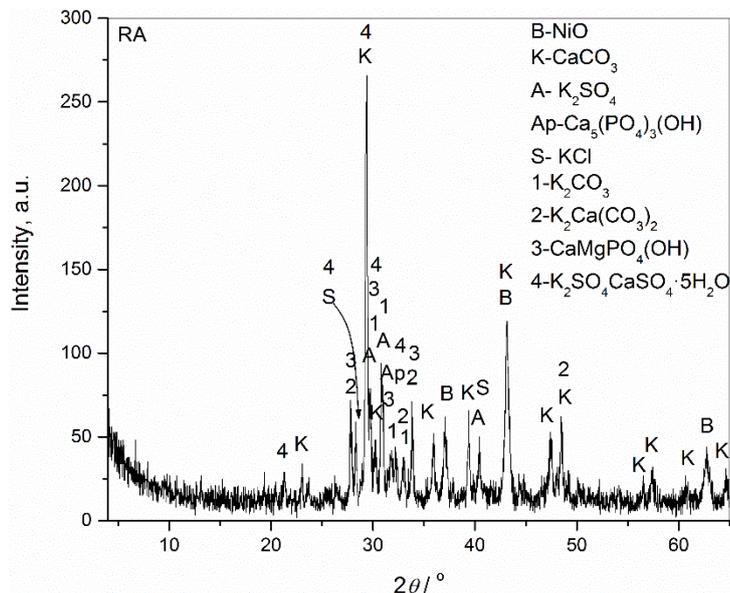


Figure 2. XRD analysis of raw ash (RA)

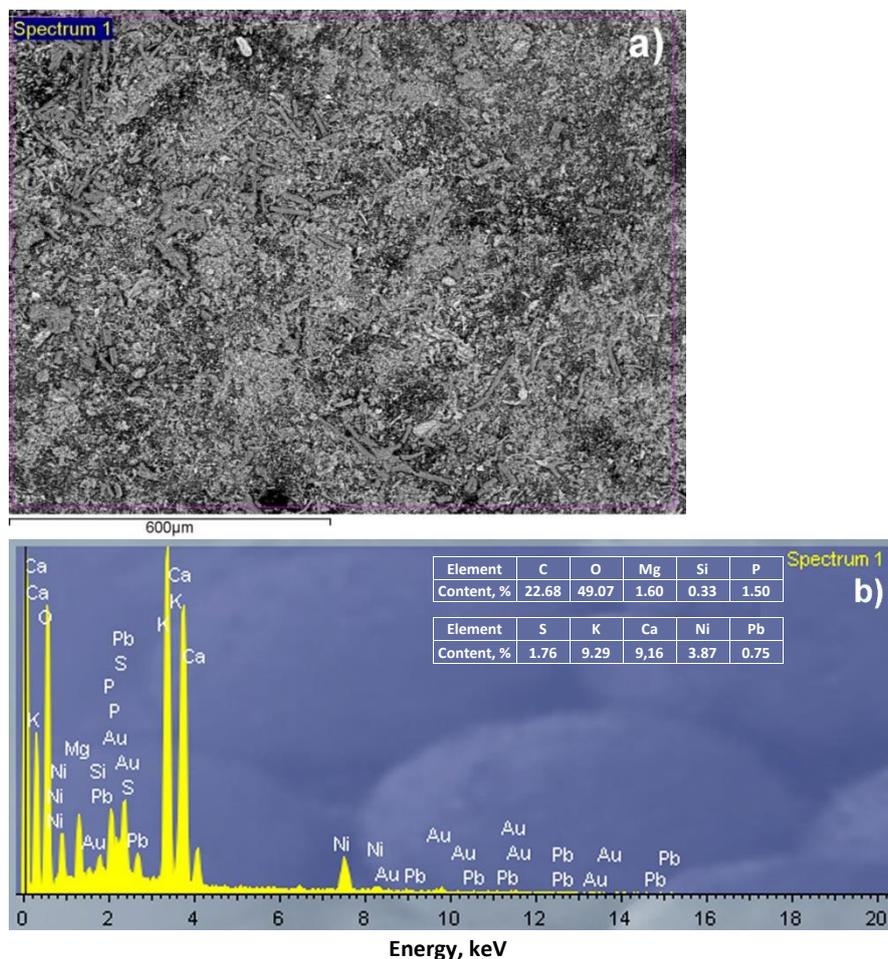


Figure 3. SEM-EDS analysis of the RA surface: (a) SEM micrograph, (b) the corresponding EDS spectrum together with numerical values

### 3. 2. Ash washing and potassium removal

Element concentrations determined in ash after the first and second washing step (WA-1 and WA-2) and in the corresponding effluents are shown in Table 2. The amount of water required for washing is determined by the dry



weight of the ash, as it was found that the optimum solid-liquid ratio is 1:4. Potassium removal is most efficient at this ratio, as solid-liquid contact is reduced at higher solid phase values [13,18].

Table 2. Element contents in ash after the first (WA-1) and second washing step (WA-2) and in the corresponding effluents (EF-1 and EF-2, respectively)

Sample	Ca	Fe	K	Mg	Ni
	Content, g kg <sup>-1</sup>				
WA-1	250 ± 42.6	2.420 ± 0.13	185 ± 13.5	57.5 ± 7.1	47.5 ± 5.3
WA-2	217 ± 28.3	2.220 ± 0.800	38.5 ± 3.6	48.7 ± 5.2	40 ± 6.9
Sample	Content, mg dm <sup>-3</sup>				
	Ca	Fe	K	Mg	Ni
EF-1	11.0 ± 0.700	0.58 ± 0.050	27100 ± 285	11.7 ± 6.30	1.20 ± 0.100
EF-2	9.50 ± 0.120	0.230 ± 0.030	5670 ± 72	26.3 ± 8.90	0.700 ± 0.060

By comparing the data for RA (Table 1) and washed ash samples (Table 2), it can be noticed that concentrations of all elements in the ash increased upon first washing. This is due to the massive weight loss in the ash due to leaching of K<sub>2</sub>CO<sub>3</sub>, which is confirmed by a significant amount of potassium determined in the effluent WA-1 (Table 1). In specific, the weight loss after the first washing step was about 25 % after the first washing step and about further 10 % after the second washing step.

Expected reduction in concentrations of all investigated elements in the ashes is noted after the second washing step. Since most of potassium was removed during the first step, alkalinity of the effluent was lowered, allowing for the subsequent extraction of magnesium and calcium.

### 3. 3. Leaching and purification

According to a previously reported study [36], optimal parameters for leaching Ni from *O. muralis* ashes to obtain a yield of Ni in the leachate of 10.2 g dm<sup>-3</sup> were: the temperature of 95°C in 1.9M sulfuric acid for 240 min at the solid-liquid ratio of 150 g dm<sup>-3</sup>. This was confirmed by Zhang [13], who reported the temperature of 70°C as sufficient, but to avoid the use of concentrated acid, leaching at 95°C is more efficient. In addition to the indicated temperature, the optimum leaching conditions are an ash-to-acid solid-liquid mass ratio of 1:9, process duration of 2 h, and 2M sulfuric acid concentration [13]. Moreover, previous studies on the leaching kinetics of ashes of a hyperaccumulating plant *Alyssum murale* by sulfuric acid [37] showed that extraction of Ni was mainly affected by the temperature and acid concentration and that the extraction rate of all the elements analyzed (Ca, Fe, K, Mg, Mn, Ni, P) was neither limited nor controlled by external film diffusion.

After the second washing and drying, and before leaching, in the present work the solid-liquid ration of 1:9 mass ratio was adopted according to the procedure. During the leaching process, the Ni content in the ash decreased from ~40 to ~4 g kg<sup>-1</sup>, as shown in Table 3.

Table 3. Element contents in washed ashes after leaching (SW), leachate before purification (L1), and leachate after purification (L2)

Sample	Ca	Fe	K	Mg	Ni
	Content, g kg <sup>-1</sup>				
SW	132.0 ± 37.2	0.342 ± 0.020	1.32 ± 0.40	4.60 ± 2.10	3.99 ± 0.30
Sample	Content, mg dm <sup>-3</sup>				
	Ca	Fe	K	Mg	Ni
L1	530 ± 25.8	262 ± 2.3	3430 ± 135	15200 ± 23.8	8500 ± 35.7
L2	120 ± 17.3	174 ± 1.8	1580 ± 58.6	3960 ± 74	5400 ± 44

XRD analysis of washed ashes prior leaching (WA-2) and washed ashes after leaching (SW), (Figs. 4 and 5, respectively) show that the phases present in the WA-2 sample are calcite (CaCO<sub>3</sub>), and bunsenite (NiO), while in the SW sample only, the presence of gypsum CaSO<sub>4</sub>·2H<sub>2</sub>O is detected.

Furthermore, the XRD analysis together with the SEM-EDS investigation (Fig. 6) confirm that the residue Ni at the concentration of ~4 g kg<sup>-1</sup> (Table 3), remained after leaching in the SW sample, is not chemically bound in the form of

NiO compound. Thus, we can assume that the remaining Ni is bound by a stronger ligand not dissoluble by sulfuric acid, dispersed in small undetectable amounts throughout the ash sample.

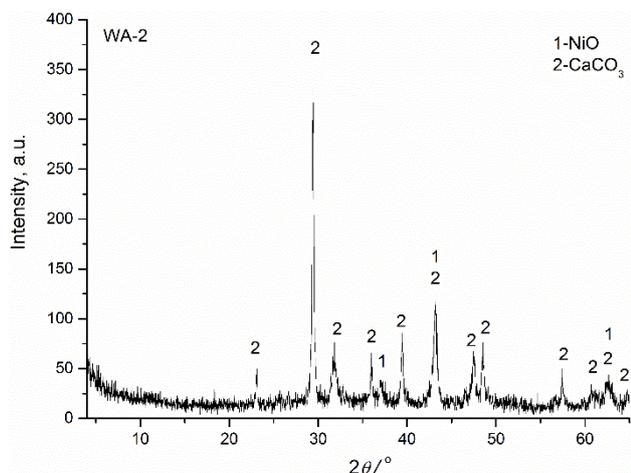


Figure 4. XRD analysis of washed ashes prior leaching (WA-2)

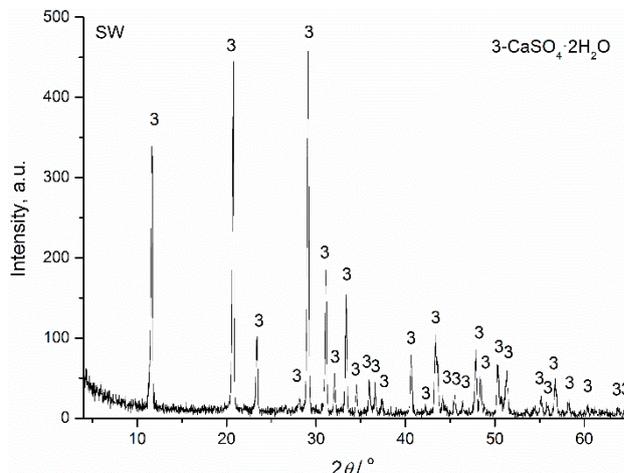


Figure 5. XRD analysis of washed ashes after leaching (SW)

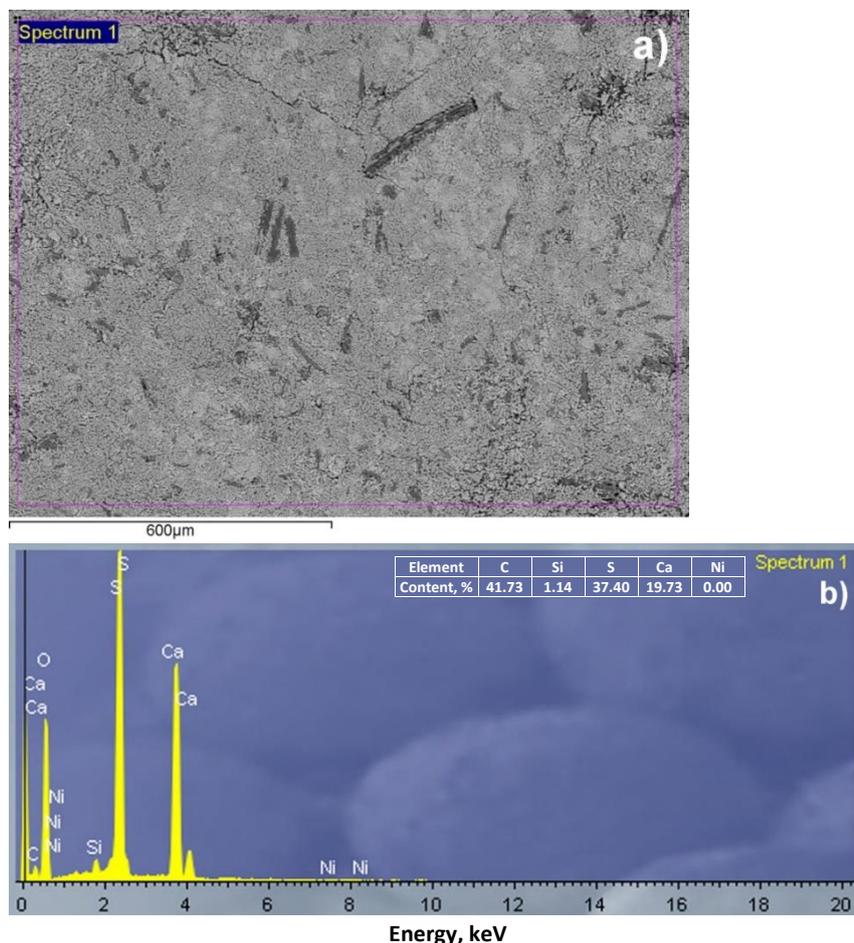


Figure 6. SEM-EDS analysis of the surface of washed ash after leaching: (a) SEM micrograph, (b) the corresponding EDS spectrum together with numerical values

After leaching and before crystallization of ANSH, the leachate had to be purified, which was carried out in two steps. In the first step, Fe was removed by precipitation in the form of Fe(OH)<sub>3</sub> due to its low solubility ( $1.5 \times 10^{-15} \text{ g dm}^{-3}$  [38]), according to the equation (1).





It was shown in literature [16] that the precipitation by  $\text{Ca}(\text{OH})_2$  is significantly more efficient as compared to the use of  $\text{NaOH}$  used previously [36]. In addition, 10 % suspension of  $\text{Ca}(\text{OH})_2$  was recommended, as there is a risk of losing a significant amount of  $\text{Ni}$  trapped in the  $\text{CaSO}_4$  precipitate when a 20 % suspension is used. As shown in Table 3, there was a decrease in  $\text{Ni}$  concentration from 8.50 to 5.40  $\text{g dm}^{-3}$  after purification. This drop can infer that there is a small amount of  $\text{Ni}$  trapped in the  $\text{CaSO}_4$  precipitate even when a 10 % suspension is used.

The next step of purification was the removal of  $\text{Mg}$  from the leachate since the presence of this element would inhibit ANSH crystallization by precipitation in the form of  $\text{MgNH}_4\text{PO}_4$ . As mentioned in the experimental section, this step was performed by adding  $\text{NaF}$  to the leachate resulting in the reaction (2).



Since the solubility of  $\text{MgF}_2$  is lower than the solubility of  $\text{NiF}_2$  (0.13  $\text{g kg}^{-1}$  vs. 25.6  $\text{g kg}^{-1}$  in water at 25 °C [39]), all  $\text{Ni}$  will remain in the leachate.  $\text{NaF}$  is added in excess of 10 % regarding the  $\text{Mg}$  content in the leachate before purification (L1, Table 3). However, the content of  $\text{Mg}$  in the obtained ANSH crystals (Table 4), suggests that the  $\text{Mg}$  removal from the leachate was not sufficient as compared to previous studies [16].

During  $\text{MgF}_2$  precipitation according to equation (2), there is also  $\text{CaF}_2$  precipitation, equation (3):



Although the  $\text{Mg}$  concentration in the leachate is higher (Table 3),  $\text{CaF}_2$  is precipitated first due to the lower solubility product ( $4.0 \times 10^{-11} \text{ mol}^3 \text{ dm}^{-9}$  for  $\text{CaF}_2$  and  $6.4 \times 10^{-9} \text{ mol}^3 \text{ dm}^{-9}$  for  $\text{MgF}_2$  at 18-25 °C [38]). Therefore, when calculating the amount of  $\text{NaF}$  required to remove  $\text{Mg}$  from the leachate, the amount of present  $\text{Ca}$  has to be considered.

After purification, the leachate may be subjected to evaporation to increase the  $\text{Ni}$  concentration. However, over-evaporation should be avoided to prevent precipitation of various dissolved  $\text{Ni}$  salts (*e.g.*  $\text{NiF}_2$ ,  $\text{NiSO}_4$ ,  $\text{Ni}(\text{OH})_2$ , *etc.*), already present in the leachate.

### 3. 4. Crystallization

To crystallize ANSH, ammonium sulfate  $(\text{NH}_4)_2\text{SO}_4$  was added to the purified leachate in 20 % excess and the suspension was heated to 60 °C. The precipitation was carried out according to the equation:



After completion of the reaction, the solution was cooled and left overnight to promote ANSH crystallization. Next, the ANSH crystals were vacuum filtered and dried. Recrystallization was performed after drying the salt crystals to increase the purity of ANSH (as suggested in literature [13]), since the solubility of ANSH in water is 175.0  $\text{g kg}^{-1}$  at 60 °C while only 19.0  $\text{g kg}^{-1}$  at 0 °C.

Chemical compositions of ANSH crystals and the solution after crystallization (SAC) are shown in Table 4.

Table 4. Chemical composition of ANSH crystals and the solution after crystallization (SAC)

Sample	As	Ca	Cd	Co	Cr	Cu	Fe	K
Content, $\text{g kg}^{-1}$								
ANSH	<0.030	<0.010	<0.030	<0.010	$0.017 \pm 0.040$	<0.010	$0.350 \pm 0.250$	$24.500 \pm 0.540$
Content, $\text{mg dm}^{-3}$								
SAC	<0.03	<0.01	<0.03	<0.01	0.017	<0.01	0.359	24.9
Content, $\text{g kg}^{-1}$								
ANSH	$20.90 \pm 0.44$	$0.016 \pm 0.004$	$0.060 \pm 0.002$	$110.00 \pm 1.04$	$0.340 \pm 0.060$	<0.010	$0.970 \pm 0.220$	
Content, $\text{mg dm}^{-3}$								
SAC	21.2	0.016	0.057	111	0.35	<0.01	0.99	

The purity of the acquired dried ANSH crystals weighing  $5.2 \pm 0.4 \text{ g}$  was 73 % ( $\text{Ni}$ :  $110.0 \pm 1.04 \text{ g kg}^{-1}$ ). This is somewhat lower than purities reported in literature of 88.8 % ( $\text{Ni}$ :  $132.0 \pm 3.0 \text{ g kg}^{-1}$ ) [36] and 99.1 % ( $\text{Ni}$ :  $149.0 \pm 1.0 \text{ g kg}^{-1}$ ) [13].

The main reason for differences in purity is the insufficient removal of Mg during the purification step, which can be easily remedied by using a higher amount of NaF by taking into account the presence of Ca in the leachate.

### 3. 5. Feasibility of Ni phytomining by *O. muralis* in Serbia

Ultramafic areas represent natural sources of Ni, which concentration is often quite higher than the established limits [40, 41], but still below the grades suitable for conventional mining. Therefore, such areas are considered particularly favorable for the development of phytomining techniques. The largest ultramafic massifs in Europe are located in Serbia [42], where suitable areas could be selected to establish phytomining plots. A wide range of nickel concentrations has been recorded in the ultramafic soils of Serbia, *i.e.* from 0.750, up to 2.300 g kg<sup>-1</sup> [43], generally mostly exceeding 1.000 g kg<sup>-1</sup>. For example, a median Ni concentration from 7 ultramafic sites in Serbia was reported as 1.330 g kg<sup>-1</sup> [44]. Even at small geographical scales, microedaphic factors can lead to significant differences in soil Ni concentrations and strongly influence the degree of Ni accumulation in some hyperaccumulating species [45]. Organic matter contents as well as the contents of macronutrients such as N, P, K, also greatly vary between the sites [43,44,46]. Therefore, appropriate agrochemical analyses are required before establishing phytomining plots. Long-term field trials in Albania [27] and studies in Austria and Greece [47] have demonstrated that phytomining could become an economically viable agricultural cropping system if the appropriate agronomic techniques are applied. However, these agronomic practices should be well adapted to regional conditions. For example, manure seems to be much less effective than inorganic fertilizers in acidic soils with low Ni mobility, while it is much more efficient in soils with high concentrations of available Ni [47]. Therefore, appropriate agronomic practices adapted to the present edaphic and climatic conditions could be proposed for selected agromining plots in Serbia after the initial examination of soil properties.

Based on the high phenotypic plasticity and Ni concentrations of up to 13.200 g kg<sup>-1</sup> detected in its leaves [6], *O. muralis*, a native metallophyte widely distributed on ultramafic substrates in Serbia, could be a suitable candidate for Ni agromining. According to the results of field trials with *O. muralis* in Albania, the optimal annual Ni yield could reach up to 100 kg ha<sup>-1</sup> [27], while in the first studies with the same species, conducted without fertilization, only 3 kg Ni ha<sup>-1</sup> were harvested [24]. Apart from *O. muralis*, several other Ni hyperaccumulators are widely distributed in Serbia, including species of the genus *Noccaea*. Although species from this genus have lower biomass production than *O. muralis*, up to 30 kg Ni ha<sup>-1</sup> was obtained in the biomass of *N. goesingensis* (Halácsy) F. K. Mey. in Austria [26]. As numerous populations of *N. kovatsii* (Heuff.) F. K. Mey. and *N. praecox* (Wulfen) F. K. Mey., phylogenetically closely related to *N. goesingensis*, have been detected in Serbia with Ni concentrations above 10.0 g kg<sup>-1</sup> [44], the potential of these autochthonous species for Ni phytomining in Serbia could be also assessed.

Life Cycle Assessment (LCA) of Ni phytomining chain was conducted for *O. murale* in Albania, identifying opportunities for improvement and showing the overall low environmental impact of the whole process [48]. LCA showed that anti-erosion practices have to be taken into account, coupled with optimizations and improvements of the Ni-recovery process, especially in terms of processing generated effluents or solid residues from the synthesis of the ANSH salts. Moreover, the possibility of using heat generated by biomass combustion and returning certain by-products from the process to the field in order to improve soil fertility could reduce the costs and increase the added value of the whole phytomining process.

In the future, after the successful technological acquisition of high-purity ANSH crystals or another valuable Ni product, an economic feasibility study of Ni phytomining in Serbia should be conducted to assess the potential environmental impact. The preliminary results of this study reveal the potential for establishing sustainable Ni phytomining plots in Serbia using native Ni hyperaccumulating species *O. murale*, based on the experiences gained in the European network of agromining field sites [18].

## 5. CONCLUSION

The average Ni content in *O. muralis* populations from ultramafic sites in West Serbia was up to 3.300 g kg<sup>-1</sup>, indicating hyperaccumulation potential and suitability for a successful Ni recovery. During combustion of the plant biomass, the weight was reduced for ~93 %, and the resulting ash was a suitable raw material for Ni extraction. After

the leaching process, crude ash yielded ANSH crystals with purity of 73 %. The purity of the recovered crystals can be further increased by adjusting the Mg removal from the leachate during the purification process before crystal precipitation.

Results of this study show the potential for implementing phytomining practices in ultramafic areas in Serbia. Moreover, by optimizing the hydrometallurgical process of ANSH extraction, it is possible to obtain salt crystals of higher purity, which would increase the economic profitability of this process. In addition, it is possible to significantly increase the Ni yield by establishing cultivation plots, where appropriate agronomic methods could be implemented.

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## SAŽETAK

### Ekstrakcija amonijum-nikl-sulfat-heksahidrata hidrometalurškim procesom iz biljke hiperakumulatora *Odontarrhena muralis* – studija slučaja iz Srbije

Branislav Marković<sup>1</sup>, Dragana Ranđelović<sup>1</sup>, Gvozden Jovanović<sup>1</sup>, Gordana Tomović<sup>2</sup>, Ksenija Jakovljević<sup>2</sup>, Tomica Mišljenović<sup>2</sup> i Miroslav Sokić<sup>1</sup>

<sup>1</sup>Institut za tehnologiju nuklearnih i drugih mineralnih sirovina, Bulevar Franša d'Eperea 86, Beograd, Srbija

<sup>2</sup>Institut za botaniku i Botanička bašta, Biološki fakultet, Univerzitet u Beogradu, Takovska 43, Beograd, Srbija

(Naučni rad)

Fitorudarenje je savremena tehnika koja koristi biljke hiperakumulatore kao vrstu bio-rude u cilju ekstrakcije metala iz njihove biomase. Hiperakumulator nikla, vrsta *Odontarrhena muralis* široko je rasprostranjena na ultramafitskim zemljištima u Srbiji i može biti potencijalno dobar izbor za primenu u fitorudarenju nikla. U ovoj studiji ispitana je efikasnost hidrometalurškog procesa regeneracije nikla iz biomase prirodnih populacija *O. muralis* putem sinteze soli nikla iz pepela biljaka u formi amonijum-nikl-sulfat heksahidrata  $\text{Ni}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$  (ANSH). Prosečan sadržaj nikla u populaciji *O. muralis* sa ultramafitskih područja u Zapadnoj Srbiji iznosio je  $3.300 \text{ g kg}^{-1}$ . Maseni prinosi ANSH kristala iz početne količine sirovog pepela je bio oko 12% prosečne čistoće 73%. Optimizacijom procesa prečišćavanja koji prethodi precipitaciji ANSH kristala, moguće je dobiti kristale povećane čistoće, što bi uvećalo ekonomsku profitabilnost ovog procesa. Rezultati ove preliminarne studije na prirodnim populacijama *O. muralis* u Srbiji pokazuju značajan potencijal za implementaciju prakse fitorudarenja kao alternativnog načina za ekstrakciju nikla sa ultramafita ovog područja.

*Ključne reči:* fitorudarenje, bio-ruda, luženje, ekstrakcija nikla