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TECHNOLOGY, COMMUNICATION AND TRANSPORT

# COBALT AND NICKEL RECOVERY FROM IRON-RICH MINE DRAIN- AGE WATER

Case study: Hitura mine site

AUTHOR:

Peetu Pesonen

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Author(s) Peetu Pesonen	
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<p>Abstract</p> <p>This thesis describes a pilot-scale trial on the Morecovery project's modular recovery system for critical raw materials (CRM), rare earth elements (REE) and base metals from mine drainage waters. The pilot-scale trial was carried out at an old Finnish nickel mine, Hitura, which was an active mine site for 45 years before being closed in late 2015.</p> <p>In the trial at Hitura, the selected metals for extraction were iron, nickel and cobalt. Iron extraction proved challenging while trying to keep as much nickel and cobalt within solution as possible for later phases of the process. Amount of co-precipitating nickel and cobalt during the iron removal process was studied in different scenarios in which process pH, volume and mixing intensity was varied. The selected process comprised two stages, where the first step was to extract iron via an oxidation-precipitation reaction, leaving nickel and cobalt in the solution, and the second step was to recover nickel and cobalt selectively. Process outputs were iron-rich sludge, nickel- and cobalt-rich sludge and purified water resulting from the purification process.</p> <p>The contents of this thesis are partly confidential and those sections are only to be distributed by the client, GTK.</p>	
<p>Keywords</p> <p>Morecovery, nickel, cobalt, Hitura, water treatment, CRM, critical raw materials, REE, rare earth elements, side streams, dissolved metals, modular recovery, base metal recovery</p>	

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## 1 INTRODUCTION

Securing critical raw material (CRM) availability is a globally important issue that is particularly relevant in terms of modern day technologies. In the project "Modular recovery process services for hydrometallurgy and water treatment" (Morecovery project), modular technology is being used for recovery of selected CRM and rare earth elements (REE) through varying processes. In the Hitura case study described in this thesis, the recovered materials were iron, nickel and cobalt. Of these only cobalt is registered as a CRM by the European Commission because of the notable probability of disruption risks. These risks arise because global production comes mostly from Democratic Republic of Congo (DRC), which has several instabilities in terms of the political situation and social effects of artisanal mining. Nickel was being monitored in the Morecovery project due to the increasing demand for this metal in advanced electronics and renewable energy technologies. Iron was recovered in the project because it impedes successful high-yield extraction of nickel and cobalt.

The Morecovery project comprised several stages, in which properties of drainage water on-site were monitored and examined. Extraction methods developed in laboratory research were upscaled to pilot scale in a modular system built inside a shipping container. The container is designed to be transported to different sites and run on-site, or by remote access. During the piloting phase of the project, different process parameters were examined, with the focus on pH, flowrate and mixing intensity.

## 2 CRITICAL RAW MATERIALS

In 2011, the European Commission drew up a list of CRM with the purpose of identifying raw materials with a high supply risk and of high economic importance, in order to secure for reliable and unhindered future access to these CRM by European industry. The list is updated by the European Commission every three years, so there are existing lists for 2011, 2014, 2017 and 2020. The 2011 list consisted of 14 CRM, but this figure has been upgraded every three years and now the list covers 30 materials (Table 1). The list is updated not only by adding more materials, but also by assessing whether the CRM currently on the list are still of high importance or high risk as regards supply. For example helium was removed from the list in 2020 because of a decline in its economic importance, but it still remains risky in terms of supply. In terms of CRM production, China is the most influential country, as it holds the majority of the global CRM supply. (COM/2020/474 final)

Table 1. European Commission list of critical raw materials in 2020 (new entries compared with 2017 are shown in bold) (modified from COM/2020/474 Final)

Antimony	Coking coal	Light Rare Earth Elements (LREE)	Platinum group metals	Tungsten
Baryte	Fluorospa	Indium	Phosphate rock	Vanadium
Beryllium	Gallium	Magnesium	Phosphorus	<b>Bauxite</b>
Bismuth	Germanium	Natural Graphite	Scandium	<b>Lithium</b>
Borate	Hafnium	Natural Rubber	Silicon metal	<b>Titanium</b>
Cobalt	Heavy Rare Earth Elements (HREE)	Niobium	Tantalum	<b>Strontium</b>

The European Commission documentation on CRM states that:

“The EU action plan for critical raw materials should:

- develop resilient value chains for EU industrial ecosystems;
- reduce dependency on primary critical raw materials through the circular use of resources, sustainable products and innovation;
- strengthen the sustainable and responsible domestic sourcing and processing of raw materials in the European Union, and
- diversify supply with sustainable and responsible sourcing from third countries,

strengthening rules-based open trade in raw materials and removing distortions to international trade.” (COM/2020/474 Final).

The European Union (EU) has set goals for extractive waste management and hopes to enhance the usage of secondary raw material sources. Benefits of secondary sources include less need for primary resources, lower import dependency, lower environmental impacts due to shorter transport distances for raw materials and lower environmental impacts on-site due to reduced amount of metal leaching. There are also financial benefits for the companies involved, as some associated costs, such as landfill tax, are reduced. There is a possibility of the EU implementing a ban on land-filling in future, which might stimulate recycling of industrial residues (Blengini et al. 2019).

## 2.1 Cobalt

Cobalt is a CRM with serious risks of disruptions in imports. DRC produces 59% of the global cobalt supply, which creates a serious issue in terms of import. The EU has an import reliance for cobalt of 86%, with 68% coming from DRC (COM/2020/474 Final). Having the majority of imports coming from DRC poses a huge political risk. According to Bloomberg (2018), DRC is considering declaring cobalt to be a strategic substance, which would more than double the export taxes from 2% to 5% and thus cause an aggressive increase in the price of cobalt. DRC also uses some unethical means for obtaining cobalt, such as artisanal mining (Figure 1), which comprises around 20% of DRC's cobalt industry (Alves et al. 2018). Some of the unethical means include the use of child labour and exposure of workers to contaminated liquids, food, air and in some cases radiation from uranium (Tsurukawa et al. 2011).



Figure 1. Artisanal mine in Democratic Republic of Congo (source: Intergovernmental forum on mining, minerals, metals and sustainable development 2017).

In addition to DRC's almost monopolitical situation, a cobalt foresight report states that uptake of LIB (Li-ion batteries) and other cobalt usages will push annual compound growth rate in cobalt demand by between 7% to 13% (Alves Dias et al. 2018). This increase would translate on average

into global consumption of 220 000 tonnes of cobalt in 2025 and 390 000 tonnes in 2030, if substitution mechanisms or alternative battery chemistries requiring less or no cobalt are not adopted. If new battery chemistries are taken up in the electric vehicle (EV) industry, cobalt consumption can be reduced by 17% by 2025 and an additional 12% by 2030. The current global cobalt production capacity is estimated to be around 160 000 tonnes per annum, which may increase to 193 000 to 237 000 tonnes per annum. The ratio of supply and demand is expected to show a deficit by 2024, but using stored resources can push the deficit further into 2025 (Alves Dias et al. 2018), although there is some deviation depending on scenario assumed (Figures 2 and 3).

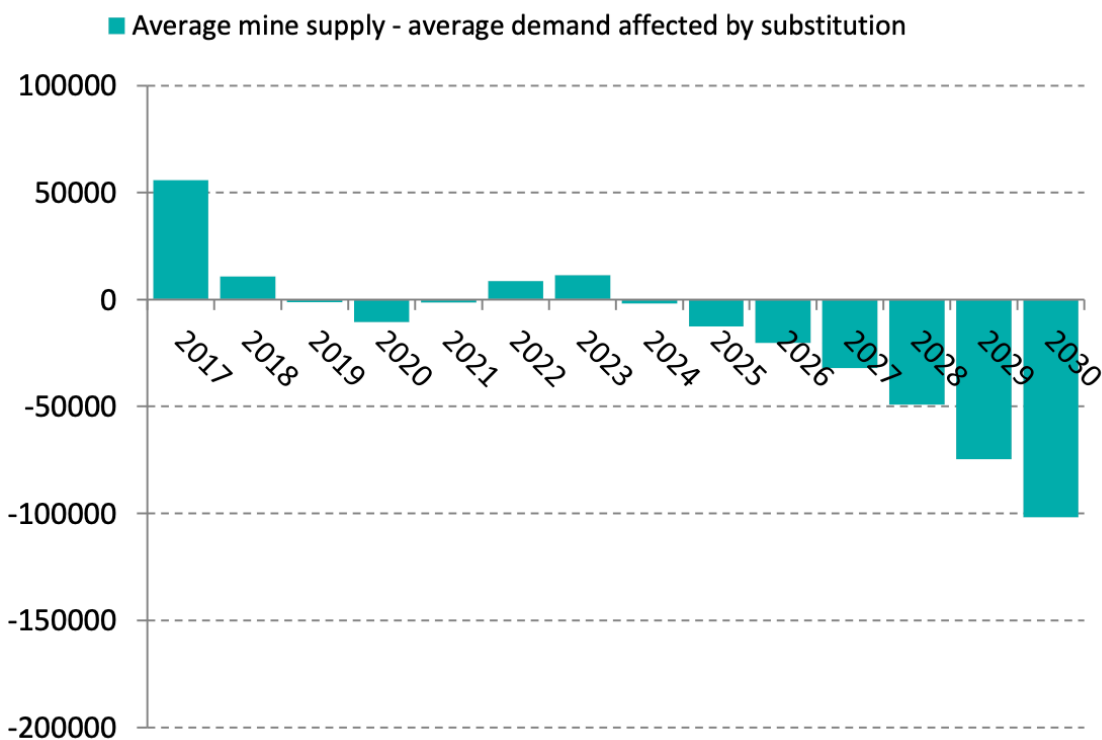


Figure 2. Projected global annual cobalt surplus/deficit (tonnes), 2017-2030 (source: Alves Dias et al. 2018).

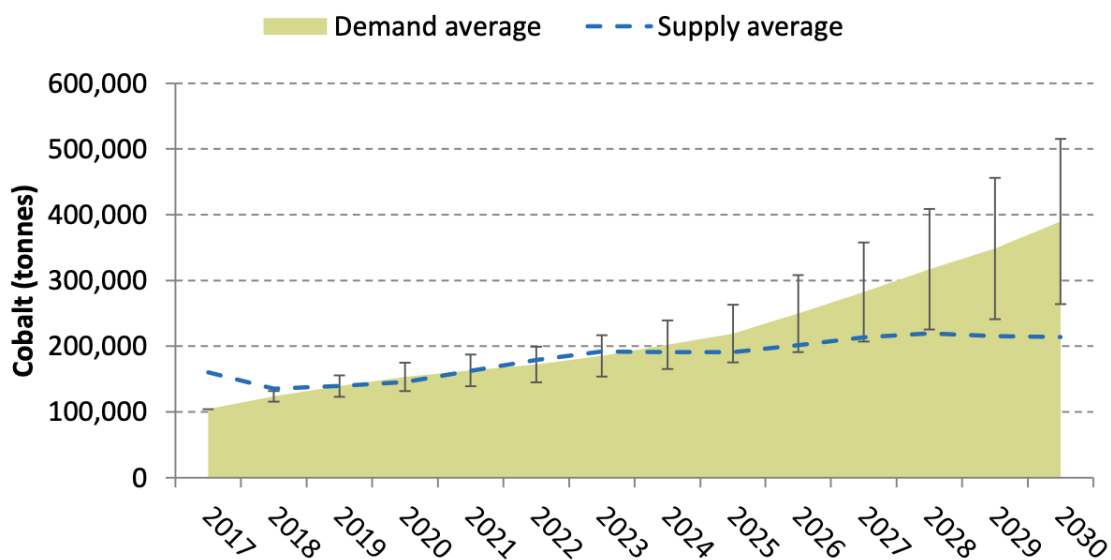


Figure 3. Projected global annual cobalt surplus/deficit 2017-2030 in average mine supply and demand scenarios (source: Alves Dias et al. 2018). *Error bars show standard deviation of demand forecasts in the scenarios.*

### 2.1.1 Nickel

Nickel has not made its way onto the CRM list yet, but may do so in future as nickel is the most probable element to replace cobalt in EV batteries, as either whole or partial substitution. Although nickel is an element of high potential and is economically important, it does not pose such a risk of disruption in terms of imported cobalt (COM/2020/474 final). Nickel supply is quite well distributed globally. In 2018, the largest nickel producer was the Philippines, which produced around 14.8% of global nickel, while the second largest were New Caledonia and Russia, each with 9.3% of total global nickel production (Garside 2020) (Figure 4).

In a recent study, nickel was used in LIB as an NMA (nickel-manganese-aluminium) mix with slightly lower specific capacity, but higher operating voltage (Wangda et al. 2020).



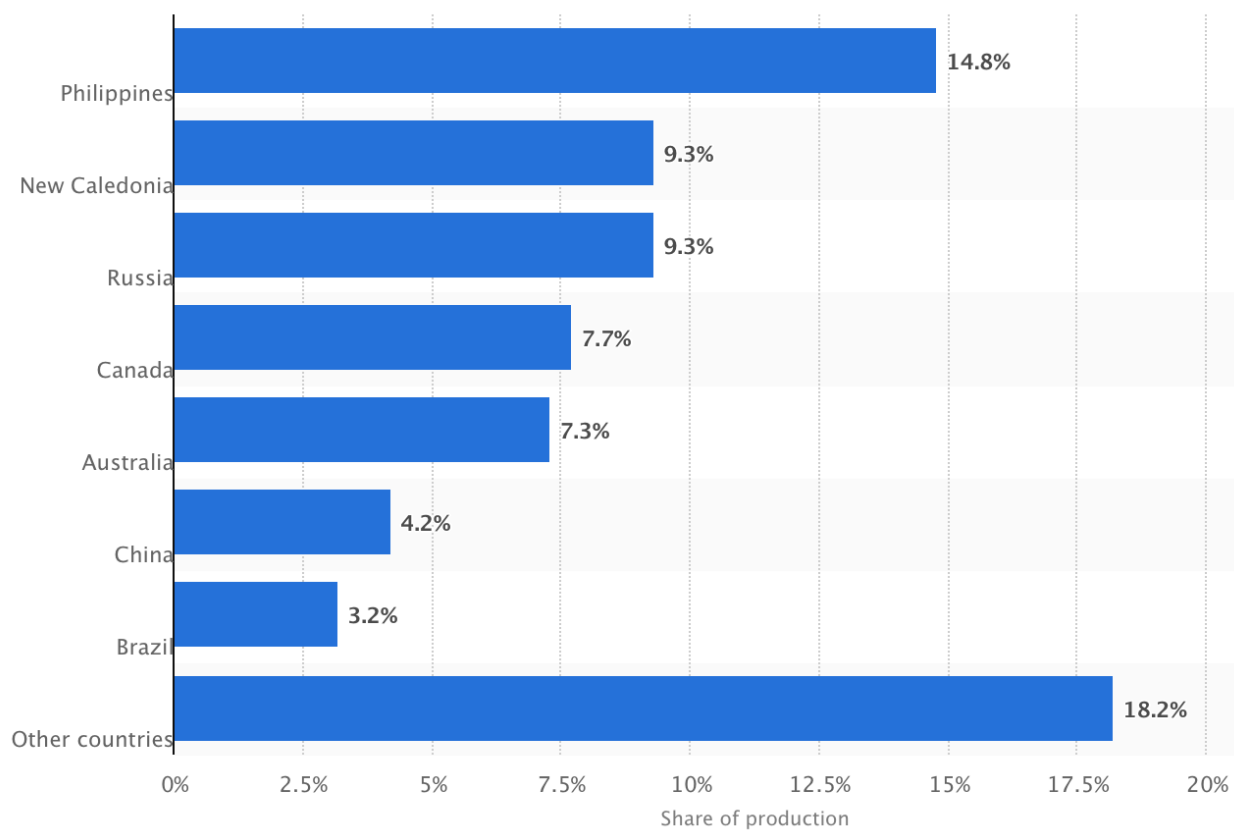


Figure 4. Distribution by country of mine production of nickel worldwide in 2018 (source: Garside 2020).

### 3 MORECOVERY PROJECT

The Morecovery project is an innovation upscaling project funded by EIT Raw Materials. The project was started in 2019 to create a cost-efficient modular service for hydrometallurgy and water treatment:

“Morecovery is a metal and mineral recovery service in the lab and/or pilot scale that allows organizations in the raw materials extractive industry to assess if their solid and liquid side streams are worth recovering metals and minerals from.” ([projects.gtk.fi/Morecovery/](http://projects.gtk.fi/Morecovery/)).

The project comprised the following eight work packages (WP) (organisation responsible in brackets):

- WP0: Go-to-market strategy (LTU Business)
- WP1: Project management (GTK)
- WP2: Professional development, learning and education (GTK)
- WP3: Update of the pilot plant and screening of potential sites (GTK, Savonia)
- WP4: Recovery piloting at laboratory scale (UEF)
- WP5: Piloting campaigns at target sites (GTK, Savonia)
- WP6: Selective metal accumulation by passive acid mine drainage remediation (UHU, CSIC)
- WP7: Marketing (GTK)

([http://projects.gtk.fi/Morecovery/research\\_activities/](http://projects.gtk.fi/Morecovery/research_activities/)).

The activities in WP3, WP4 and WP5 were the main focus in this thesis.

#### 3.1 WP3

In WP3, 18 potential pilot sites in Finland were screened (Figure 5), followed by a comprehensive assessment and ranking of their potential in terms of recovery of valuable metals and minerals from water, solid streams and waste. The assessment included closed mine sites representing different ore types, such as Särkiniemi (Ni), Kotalahti (Ni, Cu), Hammaslahti (Cu, Zn) and Hitura (Ni, Co), which were further sampled (Szlachta 2020).

During screening, samples were collected from a stream or pool of drainage water at the base of mine waste tailings deposits. The main focus in analysis of metal concentrations was on REE and other selected CRM. The samples with the highest potential were found to be from the jarosite-affected tailings drainage at the Hitura mine site. Therefore the Hitura mine was selected as a case for further studies in the laboratory and at pilot scale (Szlachta 2020).

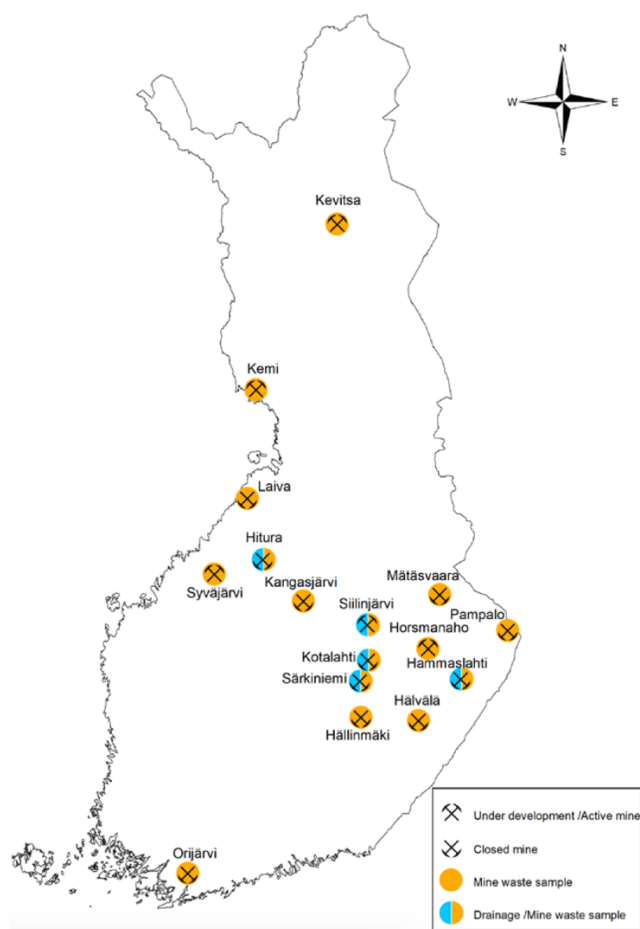


Figure 5. Location of mining sites in Finland screened for metal recovery potential in the Morecovery project (source: Karlsson 2020).

Hitura was discovered in 1961 and started as an active mine site in 1965. After 45 years of mining, the last owner closed the mine at the end of 2015. The resources mined at Hitura were mostly nickel and copper, while cobalt was mined as a side-product. Total production during the active life of the Hitura mine amounted to 92 623.39 t of nickel, 35 683.73 t of copper and 544.25 t of cobalt, i.e. nickel was the mineral produced in the largest amounts at the Hitura mine. The amount of mining waste from mineral excavation was in total 11 998 394 t. ([http://tupa.gtk.fi/karttasovellus/mdae/raportti/38\\_Hitura.pdf](http://tupa.gtk.fi/karttasovellus/mdae/raportti/38_Hitura.pdf))

Host rocks for minerals in Hitura are Iherzolite, mica gneiss, amphibolite and serpentinite, of which serpentinite has the highest composition of minerals (Table 2) ([http://tupa.gtk.fi/karttasovellus/mdae/raportti/38\\_Hitura.pdf](http://tupa.gtk.fi/karttasovellus/mdae/raportti/38_Hitura.pdf))

Table 2. Average chemical composition (wt-%) of ore types in the Hitura mine (source: Isohanni et al. 1985)

Ore type (disseminated ores)	N	D(g/cm <sup>3</sup> )	mt	Ni	Cu	Co	Fe	S	Ni <sup>S</sup>	Cu <sup>S</sup>	Co <sup>S</sup>	Fe <sup>S</sup>	S <sup>S</sup>
wall rock mica gneiss	28	2.84	0.34	0.06	0.03	0.005	4.74	2.96	0.63	0.56	0.05	57.94	40.84
marginal ore in amphibole-rock	13	2.92	0.62	0.49	0.09	0.02	7.02	4.59	6.27	1.79	0.24	55.39	36.46
marginal ore in serpentinite	27	2.71	4.89	0.65	0.17	0.03	5.01	3.38	7.97	2.22	0.34	53.79	35.37
margin of the serpentinite core	16	2.55	6.89	0.55	0.20	0.02	2.78	1.82	10.50	3.63	0.46	51.67	33.86
serpentinite core	186	2.51	6.83	0.29	0.10	0.02	1.25	0.93	12.23	3.83	0.82	47.09	35.99

N = number of samples, D = density, mt = abundance of magnetite in weight percent, Ni<sup>S</sup>, Cu<sup>S</sup>, Co<sup>S</sup>, Fe<sup>S</sup>, S<sup>S</sup> = elemental abundances calculated in 100 % sulphides.

### 3.2 WP4

In the screening in WP3, the most common minerals in Hitura side-stream leachate were determined to be iron (Fe), nickel (Ni) and cobalt (Co). The most challenging factor was to extract iron from the leachate while trying to keep as much nickel and cobalt in solution as possible. In WP4, a two-stage method for recovery of nickel and cobalt from the Fe-rich drainage water of Hitura was developed in the laboratory by UEF. The first step was to extract iron via an oxidation-precipitation reaction, leaving nickel and cobalt in the solution. In the second step, nickel and cobalt were recovered selectively. Process outputs were Fe-rich sludge, Ni-Co-rich sludge and purified water resulting from the purification process (Szlachta 2020).

In WP4, UEF also experimented with different laboratory-scale processes to find suitable means to extract high yields of nickel and cobalt by employing processes used in water treatment. The chosen methods were further optimised at pilot scale within WP5.

### 3.3 WP5

In WP5, the Morecovery project transitioned to a pilot-scale study at the Hitura mine site. A set of sea containers were used for transport and on-site usage (Figure 6). At the Hitura site, there was long container (12 m) for processes and a regular container for storing maintenance equipment and energy production. Inside the long container, there was a water treatment-based process to extract iron, nickel and cobalt from mine waste tailings, with a treatment capacity of up to 1 m<sup>3</sup>/hour. Tests on nickel and cobalt recovery were carried out to determine the best dosing amounts of the required chemicals, retention time and mixing intensity, and to assess the effects of up-scaling the process (Szlachta 2020).



Figure 6. Morecovery containers being set up at the Hitura mine site (source: Pesonen 2020).

## 4 LITERATURE EXAMPLES OF EXTRACTION PROCESSES

### 4.1 Removal of iron from acidic leachate liquor of lateritic nickel ore by goethite precipitate

A study by Yongfeng et al. (2010) examined possibilities for removing iron from Ni-rich liquor on laboratory scale, while minimising the amounts of nickel precipitating. The study used laterite ore containing large amounts of iron and aluminium (Table 3). Liquor was extracted from the laterite ore by acid leaching and the amounts of iron and nickel were analysed. The concentration of nickel was found to be  $0.63 \text{ g/L}^{-1}$  and that of iron was  $14.0 \text{ g/L}^{-1}$  (in ferrous form).

That study identified a problem with high Fe/Ni ratio in solution, which could result in excessive losses of cobalt and nickel during iron removal. The removal process comprised reducing iron-bearing solution into the ferrous state ( $\text{Fe}^{2+}$ ) and then oxidising it in air to precipitate iron as goethite (FeO). During the study, it was found that iron precipitate was in goethite form at pH values of 6.0 or lower, while higher pH resulted in decreasing crystallite size. The study also found that when the pH value was 3.0 or higher, larger amounts of nickel were lost from the process (Yongfeng et al. 2010) (Table 4).

Table 3. Chemical composition of laterite ore (mass, %) (source: Yongfeng et al. 2010)

Fe	Ni	Co	Mg	Ca	Al	Mn	SiO <sub>2</sub>
48.82	1.03	0.137	0.485	0.592	3.46	1.09	2.47

Table 4. Effects of pH value and Fe/Ni ratio on nickel losses during the iron precipitation process (source: Yongfeng et al. 2010)

pH value	Fe/Ni ratio <sup>a</sup>	Ni in residue/%	Loss of Ni/%
2.5-3.0	22.2	0.112	4.12
3.0-4.0	22.2	0.467	15.9
7.0-8.0	22.2	2.08	61.6
2.5-3.0	11.1	0.119	2.09
2.5-3.0	5.54	0.151	1.41

<sup>a</sup> Refer to the concentration ratio in the initial liquor.

#### 4.2 Process development for recovery of rare earth elements and critical metals from acidic mine leachate

A recent study examined acid mine leachate (AML) as a source of REE and other critical elements used in the manufacture of advanced electronics and renewable energy technologies. In one part of the study, they used leachate with a starting pH of 2.70, which was incrementally increased, and the content and distribution of metals after each increment were determined. After leachate pH had risen to 4.56, precipitated ferric ions were removed using a centrifuge. The percentage of metals, excluding REE, is shown on a logarithmic scale in Figure 7a and the distribution of metals in the solution is shown in Figure 7b. According to the results, iron was mostly precipitated in the pH range 2.70-3.44 and increasing the pH above 4.56 did not further precipitate iron in significant amounts. Cobalt was found to precipitate between pH values of 4.56 and 9.61, with the pH range 6.79-9.61 being most effective. Nickel showed similar results, although its precipitation range was slightly higher and it was also found to precipitate between pH 9.61 and 10.92 (Zhang and Honaker 2020).

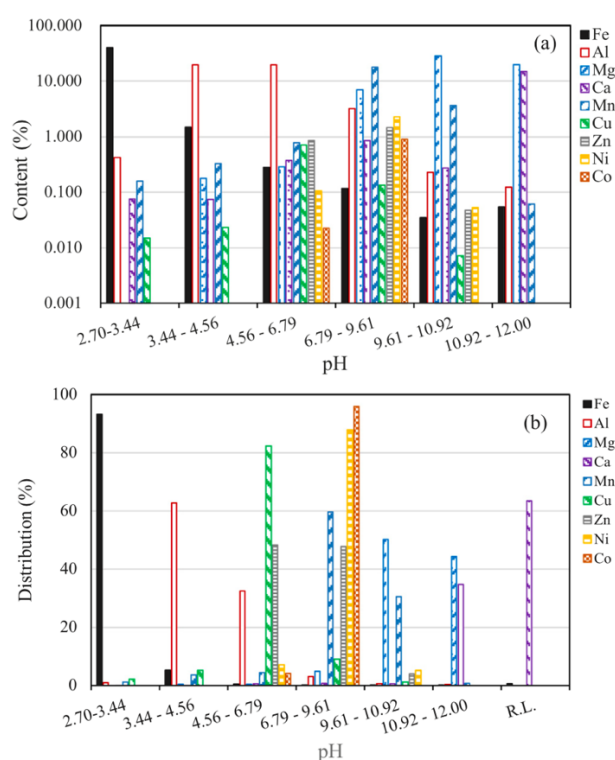


Figure 7. (a) Content (%) and (b) distribution (%) of major and trace elements in precipitate and residual liquid at different leachate pH values (source: Zhang and Honaker 2020).



## 5 PILOTING OF THE METAL RECOVERY PROCESS

The Morecovery pilot trial was carried out from 26 August to 21 October 2020. It lasted a total of eight weeks, which included start-up, pre-testing and process corrections, and the main test phase. During the campaign, Savonia was in charge of start-up and technical corrections, while Szlachta Malgorzata and Peetu Pesonen from GTK were in charge of the pre-testing and main test phase (Figure 8).



Figure 8. Peetu Pesonen working in the pilot metal recovery container at the Hitura mine site (source: Szlachta 2020).

There were several problems which prolonged the piloting time, most caused by downtimes for maintenance work at the test site. These downtimes had a restricting effect on the number of scenarios that could be tested, by limiting the time available for changing variables between scenarios. Ultimately, we managed to test one or two scenarios per day. The fastest scenarios to switch between were those with different mixing intensities.

In total, 22 different process setting scenarios were tested, by changing pH, mixing intensity, retention time and temperature. Samples were collected from influent water, after precipitation phases, from effluent water and from both sludges (Fe-rich, Ni-Co-rich). The water samples were analysed for iron, nickel and cobalt content at the Hitura site, using atomic absorption spectroscopy (AAS). Selected samples were also sent to the laboratory for analysis by inductively coupled plasma (ICP). The Fe- and Ni-Co- sludges were sent for determination of physical and chemical properties (results unavailable at the time of writing). Of these 22 scenarios, the best-case and worst-case scenarios were identified by GTK and are shown in APPENDIX 1 (Szlachta 2020).

## 6 THE PROCESS FLOWCHART IS PRESENTED IN APPENDIX 2 (PESONEN 2020).RESULTS

The pilot study data screened by GTK (APPENDIX 1) were made available for this thesis, including the best-case and worst-case scenarios. All 22 scenarios had issues with co-precipitation. In the worst-case scenario, large amounts of nickel and cobalt were lost even after the Ni-Co precipitation phase, while in the best-case scenario over 96% of nickel and cobalt were removed during the whole process (APPENDIX 3).

However, these results are associated with some uncertainty, as they were calculated based on the concentrations left in water after removing the precipitated sludge. More accurate results will be available after the sludge samples have been analysed to show the actual amounts of metals present in sludge. During testing, it was observed that some sedimentation was occurring in the precipitation tanks and it is possible that this sediment contained some of the removed elements.

It is also possible that the piloting period had some effect on the results, as the pilot test container did not have water temperature control. Water temperature can be a major factor affecting the efficiency of the extraction process, as increasing temperature greatly enhances the precipitation percentage of nickel hydroxide (Figure 9). The piloting time was only eight weeks and year-round piloting could give different results, owing to seasonal changes in water temperature and possibly even changes in mine tailings seepage composition and in metal concentrations in influent water.

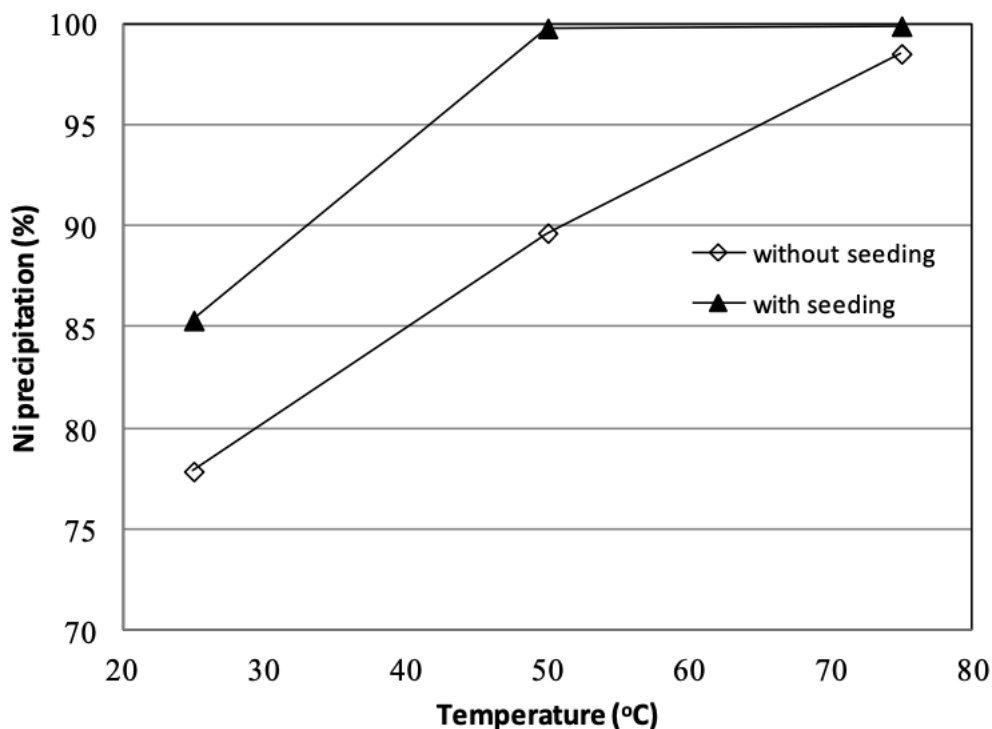


Figure 9. Plot of percentage nickel precipitation as a function of temperature at a constant pH of 7 after one hour of stirring (source: Mubarok et al. 2013).



## 7 SUMMARY

Within the Morecovery project, an eight-week pilot trial (26 August-21 October 2020) was conducted at the Hitura mine in Finland. During the trial period, 22 different metal recovery process scenarios were tested. The results showed how the actual process should be run in terms of pH, mixing intensity and retention time when taken into large-scale use at the Hitura site.

The two-step metal recovery process developed in the Morecovery project worked well on pilot scale, as iron was successfully precipitated and nickel and cobalt could then be collected. However, there were still some issues with losses of nickel and cobalt during the iron precipitation phase. Chemical analysis of the sludges obtained in the process, including analysis of the composition, are still ongoing, so more reliable conclusions can be drawn when all data from the pilot trial are available. The Morecovery project should also try running the pilot in different seasons and collecting data on the composition of the influent tailings drainage water and on the effects of seasonal changes in water temperature.

## ACKNOWLEDGEMENTS

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