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# Geochemical and mineralogical characterization of mine tailings at the Rautuvaara mine site and aspects to environmental conditions and resource potential

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## Abstract

Mining industry generates a significant amount of waste including waste rock and tailings. The disposal of mine tailings has environmental impacts, such as the releasing of heavy metals to surface and underground waters. Therefore, adequate rehabilitation of mining waste storage facilities is essential. Abandoned tailings ponds may contain significant amounts of valuable minerals, including critical raw materials, and offer opportunities as secondary mineral resources. In this study geochemical and mineralogical characterization were made for the diverse mine tailings of the Rautuvaara tailings pond which was the final disposal site for different ore deposits. The samples were collected from two different locations in the tailings pond, preconcentrated and analysed with several methods including PSA, XRD, FE-SEM, EPMA, pXRF, WD-XRF and AAS. The geochemical results indicate substantially elevated Cu, As, Ni and Zn concentrations in the tailings. Mineralogical investigations revealed that the tailings contain valuable minerals such as gold, cobaltite, and W-bearing rutile. The last could be used as an indicator mineral in tailings classification and possibly also in future ore exploration. The study of secondary mineralogy revealed that the most weathered top layers of the tailings show secondary alteration rims on the surfaces of mineral particles, and the enrichment of As and Ni in the Fe- and Mn-oxide minerals.

Keywords: mine tailings, mining waste, characterization, secondary mineralization, sulphide ore, ore exploration

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# 1. Introduction

Tailings are primarily mining waste formed in the ore enrichment processes and consist mainly of gangue minerals. They are commonly deposited from slurry-feed into tailings ponds. Sulphide minerals, such as pyrite and pyrrhotite tend to oxidize under the atmospheric conditions which may lead to the release of harmful elements containing acidic waters, often called acid mine drainage (AMD) (e.g., Nordstrom & Alpers 1999). Therefore, environmental properties and a long-term behaviour of the tailings' materials need to be characterized. The characterization of tailings refers to studies that aim to describe the chemical, physical and geotechnical features of tailings in short, medium, and long terms. These studies are used to guide the siting, dumping methods and possible utilization of tailings and other mining wastes (e.g., Amacher & Brown 2000; Kauppila et al. 2011).

Conventionally, the characterization of tailings is used to evaluate and predict their potential AMD-output, metal dissolution, and other environmental impacts. Therefore, different mineralogical and geochemical determinations from tailings' material must be studied when assessing the potential environmental impacts of the tailings. Geochemical examination traces the general chemical composition of the solids and waters concerning the occurrence of hazardous elements (e.g., Plumlee & Logsdon 1999; Smith & Huyck 1999; Jamieson et al. 2015; Parviainen 2009). The focus of the mineralogical characterization of tailings is usually on the study of sulphide mineral composition, the analysis of the proportions of the acid forming and neutralizing minerals, and a further classification of Fe-sulphide, other sulphide and non-sulphide wastes (e.g., Plumlee 1999; Kauppila et al. 2011). In addition, the mineral grain size, shape, degree of weathering and the occurrence of secondary minerals (weathering products) are also normally studied (Amacher & Brown 2000; Lottermoser 2010).

In recent years, another purpose of mine waste characterization has emerged. In the past, it was common that valuable minerals were dumped into tailings due to poorly developed mineral processing techniques, low market prices and negligible applications for some raw materials. The same characterization methods used in environmental studies can also be applied to valuable mineral potential evaluations. This type of studies has been particularly emphasized since the EU has defined historical tailings as the secondary raw material resources in the act of economic autonomy enhancement (e.g., Ladenberger et al. 2018; Markovaara-Koivisto et al. 2018; Parviainen et al. 2020). The recent proposal for a regulation of the EU raw materials act (Proposal for a regulation of the European parliament and of the council COM(2023) 160 final) urges the member states and private operators to investigate the potential for the recovery of critical raw materials from both active and historical mining waste sites.

Most of the studies which have addressed the characterization of mine tailings have focused on the environmental issues and the AMD prediction (e.g., Kontopoulos et al. 1995; Hakkou et al. 2008; Essilfie-Dughan et al. 2012). Studies on the characterization of tailings for resource potential evaluation are more limited. Ceniceros-Gómez et al. (2018) conducted a study using mineralogical and geochemical characterization methods for the tailings from the polymetallic ore processing in Mexico. The emphasis of the study was on the identification and quantification of critical minerals for their recovery. Kuhn and Meima (2019) investigated the economic potential of historical tailings from gravity separation in northern Germany. Parviainen (2009) characterized the tailings of the Haveri Au-Cu mine in Finland and created a block model for the feasibility and recovery opportunities of Au, Cu and Co from the residues. Perhaps the most advanced reprocessing project in Finland is the Otanmäki ilmenite project. Otanmäki Mine Oy together with the cooperation partners are aiming to produce ilmenite concentrate from the tailings of the former Otanmäki Fe-V-mine. Based on the results of the comprehensive characterization studies (Ilmenite project), the ilmenite concentrate production is scheduled to start in the near future.

In this study, the mineralogical and geochemical characterization of the diverse tailings of the closed Rautuvaara iron mine's tailings pond was done in connection to two ERDF (European Regional Developing Funds) funded projects: (1) 'Utilization of biochar in dry cover material and landscaping of mine waste areas' (Biopeitto) and (2) 'Automated indicator mineral identification methods for the critical mineral exploration' (Indika). One target of the Biopeitto project was to investigate the mineralogical and geochemical composition of the diverse tailings deposited in Rautuvaara (Pietilä et al. 2020). The goal of the Indika project was to study the application of indicator minerals and research techniques in critical mineral exploration with the demonstration site at the Rautuvaara tailings pond (Sarala et al. 2019). This article summarises the mineralogical and geochemical results. The main goal is to extend knowledge and characteristics of the Rautuvaara tailings and evaluate their mineral potential.

# 2. Materials and Methods

## 2.1. Study Area

The abandoned Rautuvaara mine is located in Kolari, western Finnish Lapland. The history of the Rautuvaara tailings pond dates back to the year 1962 when the Rautaruukki Oy Company started the mining activity at Rautuvaara. The mining operation ended in 1988 but the production plant was operational until 1996. Between 1965–1988 the iron oxide copper gold-type (IOCG-type) magnetite ore deposit at Rautuvaara was mainly beneficiated at the mine but also ores from two same type ore deposits nearby, Kuervaara and Laurinoja were processed in Rautuvaara. Mineral processing methods were magnetic separation for magnetite and later froth flotation for Au separation (Juopperi et al. 1982). Subsequently, the operations were managed by Outokumpu Oy Company and they were continued until 1996. During the years 1988-1996, the enriched Au-Cu- ores were mined from the Laurinoja, Saattopora and Pahtavuoma deposits. In addition, the Rautuvaara processing plant processed ore material also from the smaller test mines at Juomasuo and Kirakkajuppura, both located in Kuusamo (Räisänen et al. 2015).

The surface layers at Rautuvaara represent the tailings produced during the operations of Outokumpu Oy (Table 1). The froth flotation and gravity separation were the main processing methods for chalcopyrite and Au during the Outokumpu Oy operation time (Anttonen 1989; Lahtinen et al. 2005). It is estimated that about 9.5 Mt of tailings were deposited in the Rautuvaara tailings pond during the active period of the process plant (Räisänen et al. 2015).

The orogenic Au-Cu-deposit of Saattopora is associated with the albitite-zone of the Central Lapland belt and Au occurs as discrete grains associated with quartz carbonate veins (Korvuo 1997). The major sulphide minerals of the deposit are chalcopyrite, pyrrhotite and pyrite (Korkalo et al. 1988). According to Papunen et al. (1986) the Cu-Zn-ore bodies of Pahtavuoma represent volcanogenic massive sulphide -type (VMS-type) and the host rock is typically graphitic phyllite and, in places, metagreywacke, albite schist or skarn (calc-silicate) rock. The major ore minerals of the Cu-ores are chalcopyrite and pyrrhotite with minor sphalerite and arsenopyrite, whereas the Zn-ores are dominated by sphalerite, pyrrhotite and ilmenite with minor proportions of arsenopyrite, chalcopyrite, galena, and pyrite (Papunen et al. 1986). The orogenic Au-Co-deposit of Juomasuo is part of Käylä-Kontinaho anticline of the Kuusamo Schist Belt (Vanhanen 1992). The ore zones can be divided into the Co- and Au-Co-ore types. The former type occurs within the quartz-chlorite rocks and the latter type primarily in the quartz-sericite-chlorite rocks (Pankka 1989). The main sulphide minerals are pyrrhotite and pyrite. Cobalt together with Au are the economic elements of the Juomasuo deposit. Cobalt occurs mostly as cobaltite (Vanhanen 2001).

Table 1. Summary of the ore deposits processed in Rautuvaara. The economic ore minerals (bold), other observed ore minerals and reported gangue minerals (italics) are summarized (Räisänen et al. 2015; Geological Survey of Finland mineral deposit database (deposit reports 385, 506, 462 and 368). The disposal years and the quantities of tailings deposited are also shown.

| Ore Deposit | **Rautuvaara** | **Saattopora** | **Laurinoja** | **Pahtavuoma** | **Juomasuo** |
|---|---|---|---|---|---|
| Geological references | e.g., Papunen et al.,1986 | e.g., Korvuo, 1997 | e.g., Hiltunen, 1992 | e.g., Korvuo, 1997 | e.g., Vanhanen, 1992, 2001 |
| Ore type | IOCG | Orogenic Au | IOCG | VMS | Orogenic Au |
| Host rocks | Mafic volcanic rock | Graphite phyllite, quartz vein, intermediate volcanic rock | Diorite, hornblende-diopside metasomatic rock, magnetite metasomatic rock | Skarn, black schist, mica schist, greywacke | Sericite quartzite, mafic volcanic rock, intermediate volcanic rock, felsic volcanic rock, silicate-siltstone |
| Operator | Rautaruukki Oy | Outokumpu Oy | Outokumpu Oy | Outokumpu Oy | Outokumpu Oy |
| Years of disposal | 1962–1988 | 1989–1995 | 1978–1986 & 1989–1990 | 1992–(1993) | 1993 |
| Deposited mass, ton | 4 560 000 | 2 050 816 | 2 300 000 & 137 209 | 263 194 | 16 653 |
| Minerals | **Chalcopyrite** | **Chalcopyrite** | **Chalcopyrite** | **Chalcopyrite** | **Cobaltite** |
| | **Magnetite** | **Gold** | **Magnetite** | Pyrrhotite | **Gold** |
| | Pyrrhotite | Pyrrhotite | **Gold** | Molybdenite | Pyrite |
| | Pyrite | Pyrite | Molybdenite | Sphalerite | Pyrrhotite |
| | *Albite* | Gersdorffite | Pyrrhotite | Uraninite | Chalcopyrite |
| | *Amphibole* | Nickeline | Pyrite | Argentopentlandite | Molybdenite |
| | *Biotite* | Pentlandite | Tellurides | Arsenopyrite | Altaite |
| | *Diopside* | Tellurides | Uraninite | Gersdorffite | Calaverite |
| | *Epidote* | Tucekite | *Albite* | Ilmenite | Ilmenite |
| | *Garnet* | Uraninite | *Allanite* | Cobaltpentlandite | Cobaltpentlandite |
| | *Scapolite* | Bismuthinite | *Ambhibole* | Cobaltite | Linnaeite |
| | | *Albite* | *Apatite* | Gold | Galena |
| | | *Ankerite* | *Biotite* | Galena | Magnetite |
| | | *Dolomite* | *Calcite* | Mackinawite | Tellurobismuthite |
| | | *Graphite* | *Diopside* | Marcasite | Uraninite |
| | | *Quartz* | *Epidote* | Nickeline | Melonite |
| | | *Rutile* | *Garnet* | Pyrite | Pentlandite |
| | | *Tourmaline* | *Microcline* | Tucekite | Scheelite |
| | | | *Quartz* | *Albite* | *Albite* |
| | | | *Scapolite* | *Amphibole* | *Amphibole* |
| | | | *Titanite* | *Ankerite* | *Biotite* |
| | | | | *Calcite* | *Chlorite* |
| | | | | *Graphite* | *Dolomite* |
| | | | | *Quartz* | *Phlogopite* |
| | | | | | *Quartz* |
| | | | | | *Rutile* |
| | | | | | *Sericite* |
| | | | | | *Talc* |
| | | | | | *Titanite* |

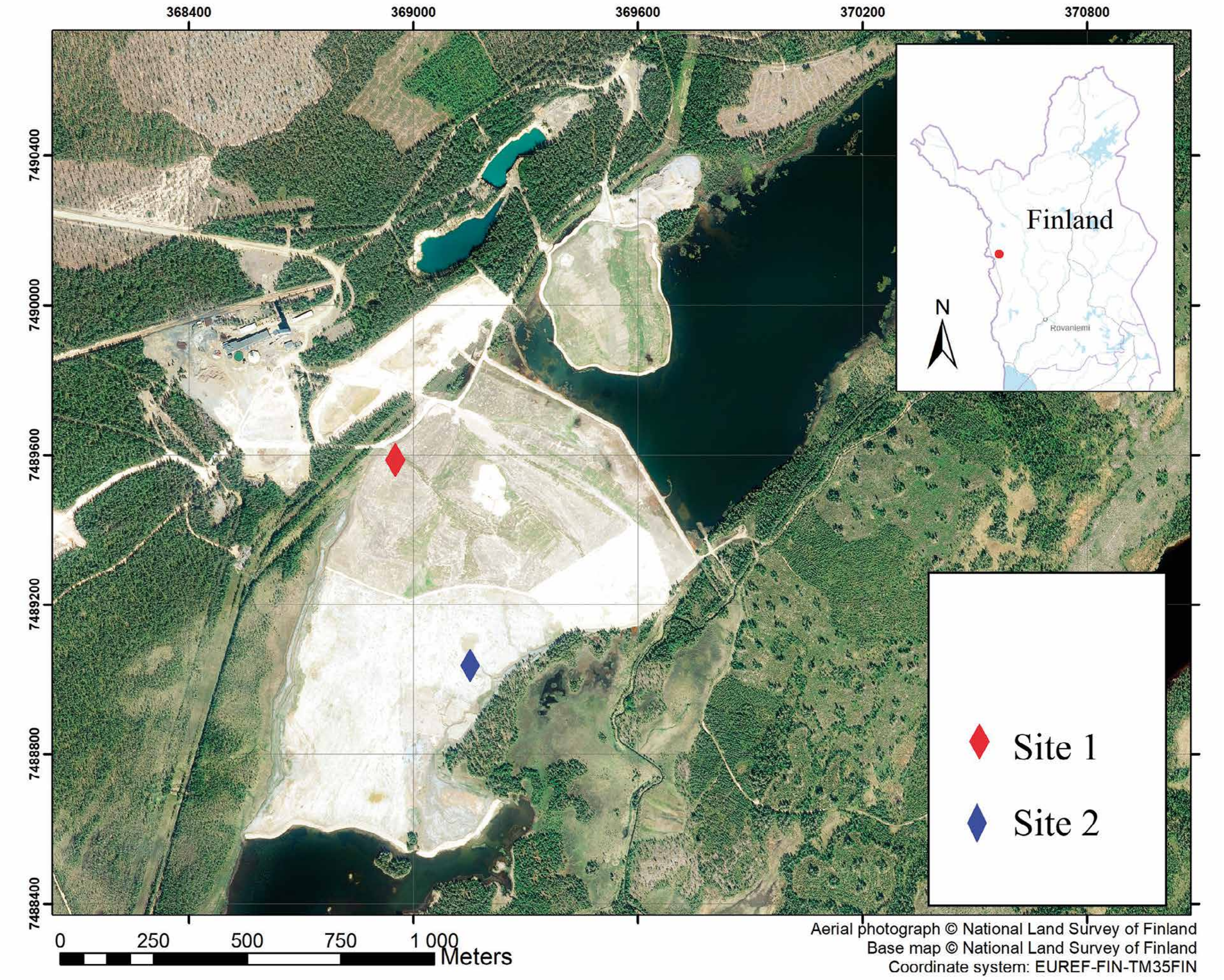

Figure 1. Sampling sites in the Rautuvaara tailings pond, Kolari.

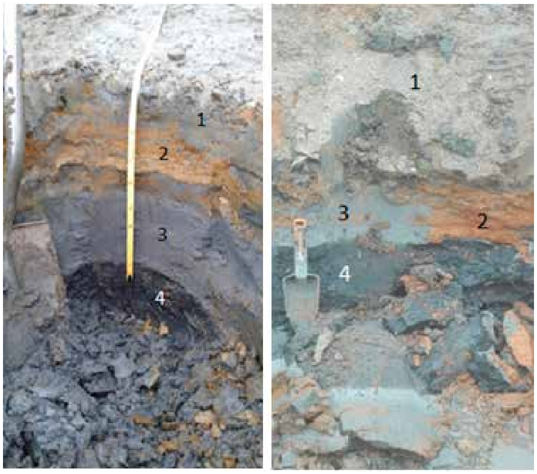

Figure 2. Sampling horizons 1-4 of the Rautuvaara tailings pond at Site 1 (left) and Site 2 (right).

The tailings studied are located on the Niesajoki riverbed. Niesajoki river was running from the northeast to southwest before the mining operations in Rautuvaara started and the river was dammed. The tailings pond with total area of ca. 100 hectare is almost 2 km long and 500 m wide (Fig. 1). Peat and till layers of varying thickness and depths beneath the tailing pond have been compacted under the mass of tailings (Räisänen et al. 2015). The Quaternary deposits of the area around the tailings pond consists mainly of sandy and gravelly till (Hirvas 1991). After the mine closure, the rehabilitation was based on the use of glaciogenic sediment together with a synthetic bentonite mat on the tailings' surface covering material. The covering procedure was completed by the end of 2018 (Mattila 2018).

## 2.2. Sampling and sample preparation

The samples were collected in 2017–2018 from two sampling pits (depth and diameters ca. 50 cm) and from the four distinctive layers in the topmost part of the tailings pond (Figs. 1 and 2). The order of the layers in the sample pits (Sites 1 and 2) starting from the top downwards is: (1) light grey layer, (2) brown and orange layer, (3) bluish grey layer, and (4) dark layer. In the following the layers are designated as follows: S1-L1 corresponds to the uppermost layer at Site 1 and the others accordingly. The style of the layering varies in different parts of the tailings pond. At Site 1, the bedding is very distinctive and continuous, but at Site 2 the layers are more lensoidal and discontinuous. Oxidation appears to be more irregular at Site 2. Furthermore, the S2-L1 is much thicker compared to the S1-L1.

In both sampling sites four samples from each four layers were taken. Three samples from each of the four layers were used in this study. The samples of Site 1 were dried and divided for bulk analyses and for the Knelson concentration. The Knelson device (KC-MD3 Model) was used to separate heavy mineral fraction for the geochemical and mineralogical analyses. The operation of the Knelson concentrator is based on the differences in particle density and an intensive whirlpool separates particles by their specific gravity (Chen et al. 2020). The following operating variables and parameters were used in this study: rotation speed 1500 rpm; centrifugal force 60 G +-2 % and fluidization water flow rate 3.5 l/min. Polished epoxy sections were prepared from the fractions (8 epoxy sections in total) for mineralogical analyses.

The samples of Site 2 were dried and split for the mineralogical and geochemical analyses (Biopeitto project). Geochemical samples were sieved into four fractions (Bulk, 250–125 µm, 125–63 µm and <63 µm) for geochemical analyses. In turn, heavy mineral separation was carried out for another part of the samples with the Knelson concentrator (with parameters described above). The treatment of the fine concentration included the heavy liquid separation with methylene iodide and lithium heteropolytungstate (LST) to separate the heavy (d > 3.3 g/cm$^3$) and middle heavy minerals (d about 2.8-3.3 g/cm$^3$). Finally, the heavy and the middle heavy mineral fractions were sieved into two size fractions (0.5-0.063 and <0.063 mm) and polished epoxy sections were prepared from all these fractions (16 epoxy sections in total) for the mineralogical analyses.

The preparation of samples from Site 2 (Indika project) included the preconcentration with a shaking table, wet sieving, low-intensity magnetic separation (LIMS), dry sieving, heavy liquid separation and high-intensity magnetic separation (HIMS) (Sarala et al. 2019). Polished epoxy sections were prepared from four fractions (8 epoxy sections in total) for the mineralogical analyses. Micro panning, ultraviolet light supported heavy mineral picking under a stereo microscope, and field emission scanning electron microscope (FE-SEM) analysis for the picked minerals were done after Knelson concentration for the split part of the sample material from Site 2 (Indika project). All the methods that were used in the study are summarized in Table 2.

Table 2. Summary of the sample preparation methods and analytical methods.

<table>
<tr><th colspan="2">Site 1 samples</th><th colspan="8">Site 2 samples (Biopeitto Project)</th><th colspan="3">Site 2 samples (Indika Project)</th></tr>
<tr><td colspan="2">Drying<br>Division</td><td colspan="8">Drying<br>Division</td><td colspan="3">Division</td></tr>
<tr><td colspan="2"></td><td colspan="5">Mineral studies</td><td colspan="3">Geochemical studies</td><td colspan="3"></td></tr>
<tr><td rowspan="5">Bulk<br>pXRF<br>FE-SEM</td><td rowspan="5">Knelson<br>pXRF<br>FE-SEM</td><td colspan="4">Knelson<br>Heavy liquid separation</td><td rowspan="5">Bulk XRD</td><td rowspan="5">Bulk<br>pXRF<br>WD-XRF</td><td rowspan="5" colspan="2">Size fractions 250-125, 125-63, <63 µm<br>AAS</td><td colspan="2" rowspan="2">Shaking table<br>Wet sieving 0.063 - 1 mm<br>LIMS<br>Dry sieving</td><td rowspan="5">Knelson<br>Micropanning<br>Stereo microscope<br>FE-SEM</td></tr>
<tr><td colspan="2">Heavy minerals</td><td colspan="2">Medium heavys</td></tr>
<tr><td rowspan="3">0.5-0.063 µm<br>FE-SEM<br>EPMA</td><td rowspan="3"><0.063 µm<br>FE-SEM<br>EPMA</td><td rowspan="3">0.5-0.063 µm<br>FE-SEM</td><td rowspan="3"><0.063 µm<br>FE-SEM</td><td>160–100 µm</td><td>100–63 µm</td></tr>
<tr><td colspan="2">Heavy liquid separation >3.3 g/cm3<br>HIMS<br>FE-SEM</td></tr>
<tr><td colspan="2"></td></tr>
</table>

## 2.3. Analytical methods

Particle size analyses (PSA) were conducted with Cilas 1190 LD particle size analyser based on the laser diffraction method with water as a medium. The dried samples were saturated by water, mixed and left to settle down to the bottom of the sample cup. After this, all the excess cleared water was decanted, and the sample was homogenized. Finally, the subsample for the actual PSA was collected with a small tip sampler through the whole depth of the sediment layer.

X-Ray diffraction (XRD) analyses to define the main mineralogy from four bulk samples of Site 2 were conducted with Rigaku SmartLab X-Ray diffractometer at the Centre for Material Analysis in the University of Oulu (CMA). The accelerating voltage was 40 kV and current 135 mA. Cu_K-beta filter and D/teX Ultra 250 detectors were used. The samples were measured in the scan range of 5° to 120° with the step width 0.02°. Scan axis was Theta/2-Theta. The whole powder pattern fitting (WPPF) was used as a phase identification and a quantification method, and finally identified using mineral deposit database of the Geological Survey of Finland (GTK) (Mineral deposit reports 602, 385, 506, 462 and 368).

Field emission scanning electron microscope (FE-SEM) analyses were conducted with JEOL FE-SEM Ultra equipment at the CMA. The SEM imaging was conducted by scanning the electron beam across the samples' surface and detecting the emitted secondary electrons (high-resolution images) or the backscatter electrons (mass contrast images). FE-SEM was equipped with the energy-dispersive X-ray detectors (EDS), electron backscatter diffraction cameras (EBSD) and the mineral liberation analyser (MLA) option for the mineralogical studies. The polished epoxy sections were prepared especially for the FE-SEM analyses. Around 5000 to 10000 mineral grains were analysed from each 33 polished sections. The unclassified results come from: (1) multiphase grains where several minerals were found in one analysed feature, (2) secondary minerals whose chemical composition does not meet the criteria of the primary minerals' library used for the mineral classification, (3) overly small crumbs of ambiguous particles, likely scraps, and impurities from the process. The classification classes can be summarized as silicates, carbonates, oxides, phosphates, and sulphides. The MLA option and the IncaMineral software were then used for the automated mineral classification and to obtain the modal mineralogy of the tailings layers.

The mineral chemistry analyses were performed with the Electron probe microanalyzer (JEOL JXA-8530F Plus FE-EPMA) at the CMA. The analyser was equipped with a wavelength dispersive X-ray spectrometer (WDS) and EDS. An accelerating voltage of 7 kV, and a current of 15 nA were used. The matrix correction, with the PRZ methods was applied to all analyses. Because of the non-destructive nature of the FE-SEM analysis, the same polished sections (8 polished sections from the samples of Site 2) could be used for the EPMA analysis. The EPMA analysis was conducted to the map alteration of mineral grains due to oxidation. The backscattered-electron imaging helped to identify relevant targets i.e., altered mineral grains and then to perform elemental analysis using EDS.

The portable X-Ray fluorescence (pXRF) analyses for the total elemental concentrations were performed with the Oxford Instruments X-MET-8000 XRF handheld analyser. Dry subsamples of bulk sample material were used without any pre-processing for the pXRF analyses. Sample material was placed and slightly pressed into the plastic cups (diameter 3.0 cm) covered by a thin polypropylene film (Oxford Instruments Poly 4). Both the soil and mining modes were used and the average values of three separate measurements were used as a total concentration of elements (cf. Sarala 2016). The pXRF analyses, which are considered as semi-quantitative and indicative, were used to obtain the chemical compositions (total concentration) of the tailings' layers and detectable elements were Mg, Al, Si, P, S, K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Rb, Sr, Zr, Nb, Mo, Cd, Sn, Sb, Ba, Ta, W, Hg, Tl, Pb, Th, U.

Table 3. Particle size analysis results for Site 1 and 2 bulk samples.

| | Site 1 | | | | Site 2 | | | |
|---|---|---|---|---|---|---|---|---|
| Particle size [μm] | | | | | | | | |
| **Layer** | **1** | **2** | **3** | **4** | **1** | **2** | **3** | **4** |
| D80% | 54 | 22 | 38 | 63 | 45 | 98 | 43 | 33 |
| D50% | 25 | 10 | 19 | 28 | 24 | 58 | 20 | 14 |
| D20% | 7 | 3 | 6 | 7 | 8 | 18 | 7 | 5 |

The laboratory-based, wavelength dispersive X-Ray fluorescence (WD-XRF) analyses were performed with a PANalytical Axios max 4kW XRF laboratory analyser at the CMA. The subsamples of bulk sample material were analysed using briquets which were prepared by pressing a few grams of powder sample, wax and boric acid. The sample preparations and analyses were done at the CMA. Detectable elements were Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, Cr, Mn, Fe, Sc, Ni, Cu, Zn, Ga, Ge, As, Rb, Sr, Zr, Y, Ba, La, Ce, Pr, Nd, Pb, Bi, U.

The atomic absorption spectrometer (AAS) analyses were conducted with the Perkin Elmer Analyst 400 equipment using a flame atomizer method in the geochemical laboratory of Oulu Mining School. The analyses were performed for three different fractions (250 – 125 μm, 125 – 63 μm and <63 μm) from Site 2 samples. Selective sequential leaching with the reagents of hydroxylamine hydrochloride ($NH_2OH$*HCl) and aqua regia ($HNO_3$+HCl; AR) was carried out for the tailings' samples and the metal leachate solutions were then analysed with the AAS. $NH_2OH$*HCl leaching is for analysing weakly bounded metal ions adsorbed mainly in the Fe- and Mn-oxides which indicates metal ion mobilization during weathering (Li et al. 1995). The aqua regia leaching is a strong digestion method which in some cases can give near total results e.g., to determine changes in an endogenic signal caused by the alteration of mineralogy during surface weathering (Ramsey 1997). As, Cu, Fe, Mn and Ni were selected for the analysis. In addition, Au was analysed with a graphite furnace atomic absorption spectrometer (GFAAS) in the Geological Survey of Finland.

# 3. Results

## 3.1. *Grain size and mineral composition*

All the samples were found relatively fine-grained but there were some differences between the sampling sites (Table 3). The parameters D80%, D50% and D20% give the diameter of percentage with smaller particles. For instance, if the D80% is 54 μm, 80 % of the sample has a particle size of 54 μm or smaller. There is one distinctively coarse layer at both sites; S1-L1 and S2-L2. Otherwise, at Site 1 the average particle size of the layers increased downwards, a feature not characteristic for the layers at Site 2.

According to the XRD analyses, the main mineralogy of the layers S2-L1, S2-L2 and S2-L3 is quite similar (Fig. 3). In all three samples,

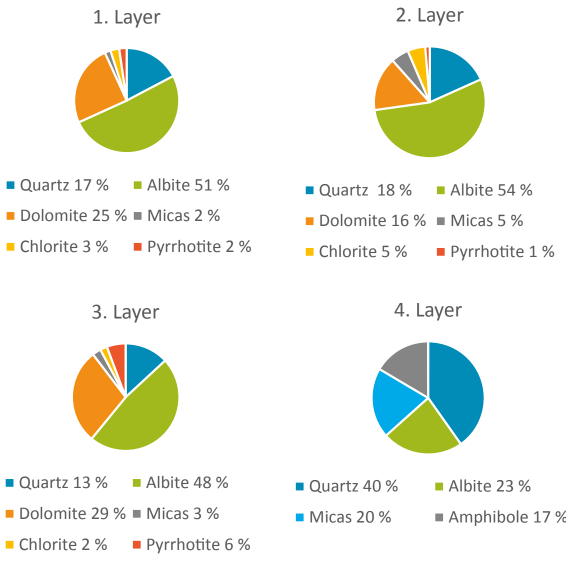

Figure 3. Main bulk mineralogy of each layer of Site 2 analysed with XRD.

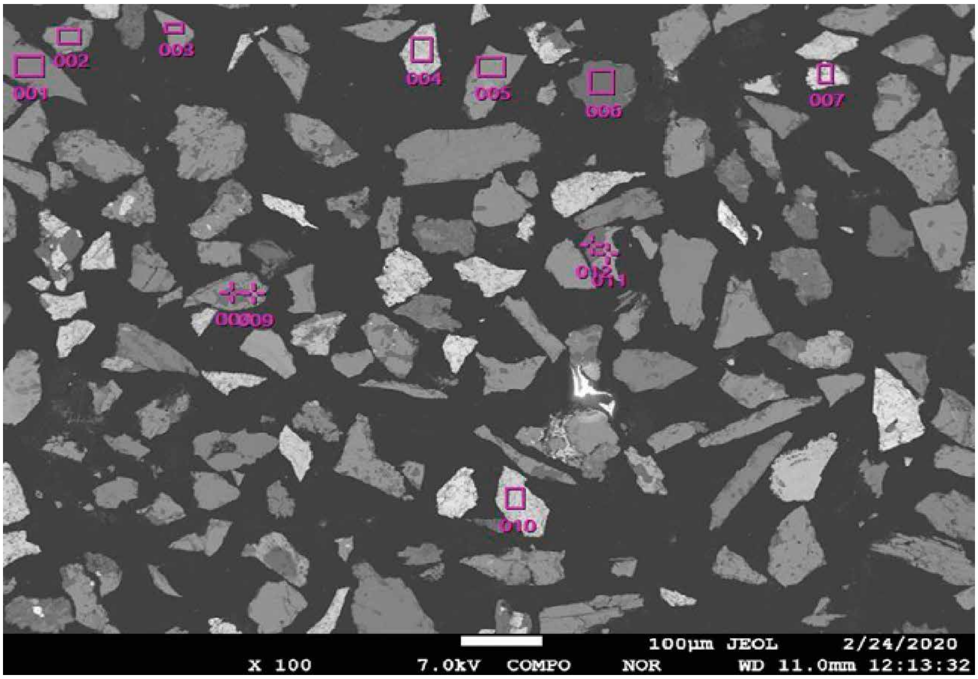

Figure 4. EPMA-EDS image of multiphase grain with W-rutile (011) and plagioclase (012) found in the layer 4 at Site 2. EDS-analysis for W-rutile gave $TiO_2$ 96.99 wt% and $WO_3$ 3.01 wt%. All the analyses are presented in the Supplementary Table 5 (Electronic Appendix A).

albite, quartz and dolomite dominate the mineral composition. The remaining part consists of micas, chlorites and pyrrhotite. The layer S2-L4 differs from the overlying units with quartz being the most abundant mineral, followed by albite, micas, and amphibole in rather even amounts.

The FE-SEM results show that the mineralogy of all samples in both sampling sites corresponds well to the mineralogy of the processed ores (Table 1; see also Supplementary Tables 1, 2, and 3 in Electronic Appendix A). Albite, quartz, micas, amphibole, and chlorite were classified into the class of other silicates. Allanite and zircon were classified separately. Together with albite and quartz, Fe-dolomite dominate the mineralogy. Fe-dolomite is evenly distributed between the layers at Site 1 while it is very rare in the layer S2-L4.

Oxides were classified into two groups: (1) rutile, ilmenite and uraninite, and (2) iron oxides including magnetite and hematite. Due to the different preconcentration methods between the Biopeitto and Indika projects, major differences in iron oxides at Site 2 can be seen. Ilmenite contents are low at both sites, but rutile quantities are constantly higher. A remarkable feature in the tailing samples is W-bearing rutile ('W-rutile') containing 1.5 – 5 wt% tungsten (Fig. 4; see also Supplementary Table 4 in Electronic Appendix A). The estimated W-rutile concentrations, based on the electron microscope imagining and heavy mineral picking as well as visual counting under stereo microscope, are 0.2-2 vol-% at Site 2, at Site 1 it barely occurs. A few grains of uraninite were found in the sample S2-L3.

Apatite and REE-minerals monazite and xenotime are included into the phosphate class. These minerals are just barely observed in samples of Site 1, but their quantity is significantly higher at Site 2. The REE-minerals seem to be more common in the layers S2-L1 and S2-L2 rather than in the bottom layers of Site 2.

The sulphides in the classifications include pyrite, pyrrhotite, chalcopyrite, arsenopyrite, sphalerite, galena, cobaltite, gersdorffite, and argentopentlandite. The iron sulphides pyrite and pyrrhotite are abundant in all samples. All layers at Site 1 nearly lack other sulphide minerals whereas the layer S2-L4 is richer in sphalerite, arsenopyrite and chalcopyrite. Minor amounts of cobaltite are present in all layers at Site 2. In addition, few grains of argentopentlandite and gersdorffite were found in the layers S2-L3 and S2-L4.

Gold and W-rutile were most significant findings from Site 2 after the micro panning and mineral picking procedure. Gold occurs as angular 0.02-0.09 mm grains, and 14-59 pieces of grains were found in each sample. Under ultraviolet light, 100-2000 pieces of fluorescence minerals were found from each concentrate. These fluorescent grains were first interpreted as scheelite, which is typical accessory mineral in Au deposits in northern Finland, but during the FE-SEM analyses they turned out to be W-bearing rutile. The rest of the micro panning heavy mineral concentrates consisted mainly of sulphides and Fe-oxides.

The microprobe (EPMA) results show the moderate degree of weathering can be observed in studied tailings (Fig. 5). Incipient alteration occurs as secondary mineral rims on the edges of mineral grains in the layer S2-L1. According to the EDS elemental analysis (see Supplementary Table 5 in Electronic Appendix A), it is assumed that the secondary accumulations are basically composed of the Fe-oxyhydroxide, such as ferrihydrite or goethite. Fe-oxyhydroxide rims occur on the surface of dolomite, albite, biotite, and Fe-sulphide grains. Fe-sulphate and Fe-hydrosulphate were occasionally found in the samples. Advanced alteration occurs in the layer S2-L2 and the secondary rims are more common and more distinct than in the layer S2-L1. In addition, Fe-sulphate rims on the sulphide mineral grains are common. In the layers S2-L3 and S2-L4 secondary rims are less common.

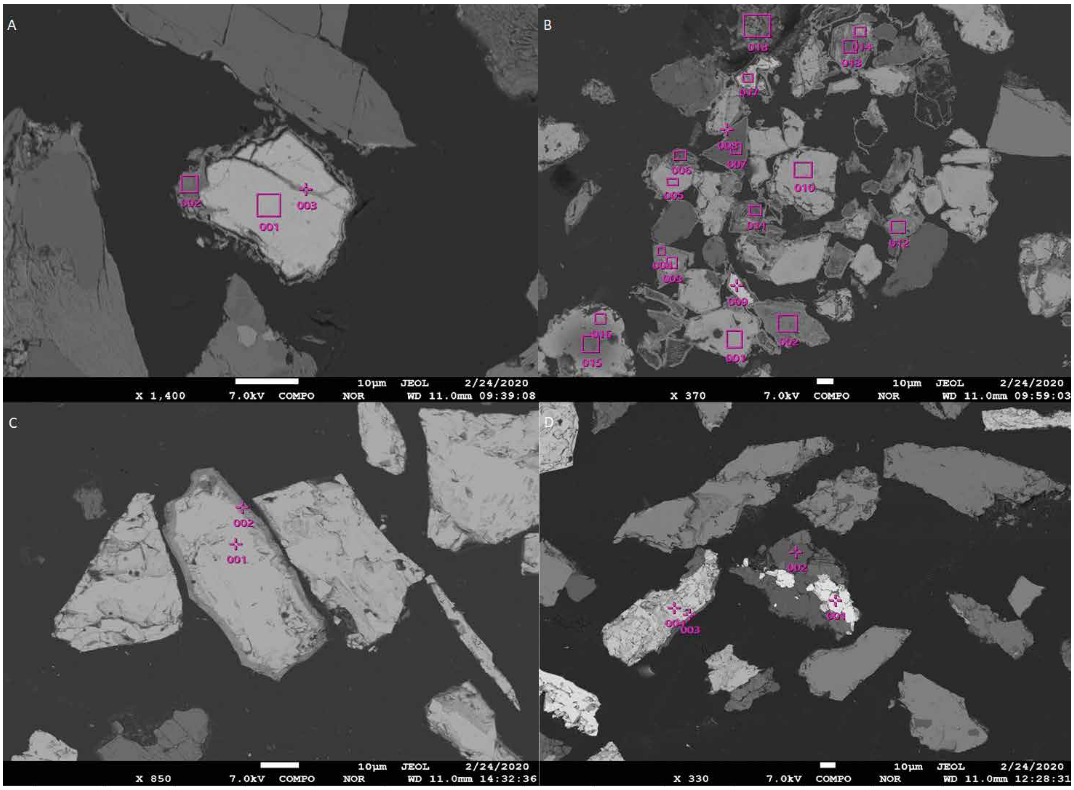

Figure 5. Backscattered-electron images of mineral grains from the layers of Site 2. a) Fe-oxyhydroxide rim (002) around Fe-sulphide grain (001) in the layer 1. Some Fe-sulphate or Fe-hydroxysulphate (003) has formed in the inner crack of the sulphide grain. b) Extensive alteration as rims around many of the grains. c) Fe-oxyhydroxide rim circles Fe-sulphide grain in the layer 3. d) Minor alteration in the layer 4. Multiphase grain shared by cobaltite (001) and quartz (002), and possible clinochlore or chamosite (003,004) of chlorite mineral group. All the analyses are presented in the Supplementary Table 5 (Electronic Appendix A)).

Table 4. Total elemental concentrations of the bulk samples of Site 2 analysed with pXRF and laboratory WD-XRF. Sodium cannot be detected with pXRF. the standard calibration of X-MET 8000 scheme does not include Sc, Ga, Ge, Y, La, Ce, Pr, and Nd. Cobolt was not analysed by WD-XRF due to the risk of contamination during sample grinding, it is not analysed. (<DL stands for under detection limit; for detection limits and reference material analyses, see Supplementary Tables 6 and 7 in Electronic Appendix A).

| | **Portable XRF** | | | | **WD-XRF** | | | |
|---|---|---|---|---|---|---|---|---|
| Layer | 1 | 2 | 3 | 4 | 1 | 2 | 3 | 4 |
| Na [%] | - | - | - | - | 3,03 | 3,51 | 3,31 | 0,95 |
| Mg [%] | 3,8 | 2,63 | 4,22 | 2,83 | 3,33 | 2,13 | 3,96 | 2,79 |
| Al [%] | 5,59 | 4,76 | 5,34 | 5,38 | 5,68 | 5,81 | 5,94 | 6,6 |
| Si [%] | 21,5 | 20,1 | 19,8 | 22,42 | 21,03 | 22,1 | 20,92 | 25,07 |
| P [%] | 0,04 | 0,01 | 0 | 0,03 | 0,05 | 0,01 | 0,04 | 0,04 |
| S [%] | 1,83 | 2,08 | 4,45 | 1,63 | 2,36 | 3,08 | 3,11 | 1,52 |
| Cl [%] | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0,12 |
| K [%] | 0,59 | 0,69 | 0,82 | 2,42 | 0,43 | 0,5 | 0,67 | 2,27 |
| Ca [%] | 7,05 | 5,63 | 7,4 | 4,2 | 6,35 | 4,94 | 6,3 | 4,17 |
| Ti [%] | 0,58 | 0,49 | 0,7 | 0,83 | 0,54 | 0,46 | 0,66 | 0,75 |
| Cr [%] | 0,05 | 0,04 | 0,07 | 0,05 | 0,02 | 0,01 | 0,03 | 0,04 |
| Mn [%] | 0,17 | 0,17 | 0,16 | 0,35 | 0,12 | 0,09 | 0,13 | 0,3 |
| Fe [%] | 12,03 | 18,45 | 12,1 | 12,55 | 8,35 | 9,3 | 8,84 | 10,35 |
| Sc [ppm] | - | - | - | - | 27 | 21 | 34 | 31 |
| Co [ppm] | 663 | 219 | 101 | 135 | - | - | - | - |
| Ni [ppm] | 1100 | 1461 | 548 | 289 | 719 | 655 | 405 | 250 |
| Cu [ppm] | 529 | 628 | 381 | 4687 | 400 | 300 | 405 | 3526 |
| Zn [ppm] | 46 | 0 | 104 | 1604 | 34 | <DL | 70 | 1365 |
| Ga [ppm] | - | - | - | - | 21 | 20 | 20 | 19 |
| Ge [ppm] | - | - | - | - | 7 | 7 | 5 | 5 |
| As [ppm] | 1556 | 643 | 331 | 2533 | 685 | 206 | 144 | 1379 |
| Rb [ppm] | 31 | 48 | 44 | 103 | 14 | 17 | 22 | 70 |
| Sr [ppm] | 117 | 117 | 97 | 137 | 52 | 31 | 43 | 84 |
| Zr [ppm] | 164 | 230 | 138 | 158 | 91 | 89 | 82 | 119 |
| Y [ppm] | - | - | - | - | 33 | 10 | 37 | 27 |
| Mo [ppm] | 0 | <DL | 0 | 0 | 7 | 6 | <DL | 6 |
| Sn [ppm] | <DL | 174 | 0 | 0 | <DL | <DL | <DL | <DL |
| Sb [ppm] | 38 | 69 | 66 | 13 | 31 | 31 | 31 | 31 |
| Ba [ppm] | 271 | 469 | 340 | 2591 | 108 | 99 | 163 | 2381 |
| La [ppm] | - | - | - | - | 51 | 53 | 39 | 27 |
| Ce [ppm] | - | - | - | - | 200 | 125 | 92 | 27 |
| Pr [ppm] | - | - | - | - | 6 | 5 | 7 | 5 |
| Nd [ppm] | - | - | - | - | 60 | 43 | 37 | 48 |
| Pb [ppm] | 22 | 15 | 17 | 132 | 14 | 12 | 12 | 112 |
| Bi [ppm] | 0 | 0 | 0 | 0 | 22 | 14 | 11 | 30 |
| U [ppm] | 27 | 37 | 8 | 0 | 23 | 18 | 8 | 5 |

## *3.2. Geochemical analyses*

Total elemental concentrations from the pXRF analyses and the WD-XRF analyses (Table 4; see also Supplementary Table 6 in Electronic Appendix A) reflect mainly the results of the mineralogical results presented above. Na, Si, Mg, Fe and Ca concentrations correspond to the major gangue minerals albite, quartz, and dolomite, respectively. The concentrations of K and Al plausible reflect the relative abundance of micas. Iron sulphides, pyrrhotite and pyrite result in relatively high concentrations of sulphur and iron in both sites. These concentrations may be exceedingly elevated as can be seen in the layers S1-L3, S1-L4, S2-L3 and S2-L4.

Elemental concentrations are relatively similar between the layers at Site 1. The most significant differences in total concentrations between Sites 1 and 2 are in Cu, Zn and As. The layer S1-L4 included minor quantities of Cu, Zn and As whereas the corresponding layer at Site 2

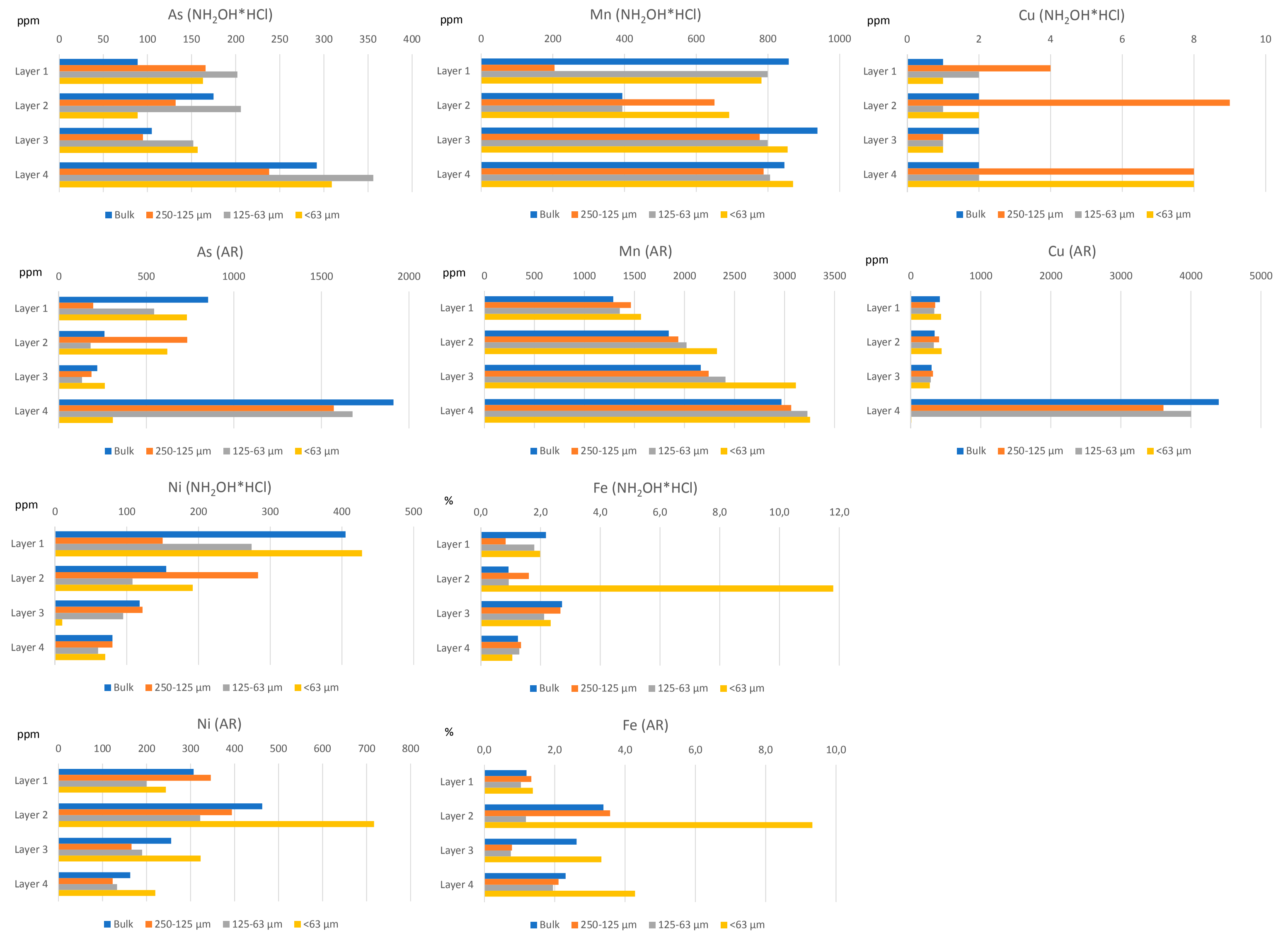

Figure 6. As, Cu, Fe, Ni, and Mn concentrations in different size fractions of each layer at Site 2. Concentrations for As, Mn and Ni expressed as ppm, and Fe as %. The samples were leached with hydroxylamine hydrochloride ($NH_2OH*HCl$) and aqua regia (AR) before analysis with AAS method.

(S2-L4) is clearly enriched with these elements. As concentrations seem to be elevated in the topmost layer at both sites. High Ba concentrations of the layer S2-L4 could indicate to the presence barium minerals (e.g., barium sulphate, baryte, Ba-bearing feldspar or mica). The anomalous chlorine concentration in the layer S2-L4 could be explained by halides formed by evaporation.

Atomic absorption spectrometry was used to obtain the concentration of elements such as As, Mn, Cu, Fe and Ni (Fig. 6). The results from samples digested by $NH_2OH*HCl$ show high concentrations of As, Mn and Ni in the upper layers at Site 2 indicating enrichment of As and Ni during weathering and their adsorption to Fe- and Mn-oxides, and possibly to the clay minerals. The Ni concentrations decrease downwards, but for As concentrations are the highest in the layer S2-L4. Furthermore, As, Mn, and Ni concentrations are higher in the small grain size fraction, particularly, in the AR digestion-based analyses. However, Cu seems not to be soluble in $NH_2OH*HCl$ and the concentrations do not have similar relationship as As, Mn, and Ni to the grain size fractions. The Cu and As concentrations from AR digestion are particularly high in the layer S2-L4. Accordingly, the As, Cu and Mn concentrations in the XRF results are much higher in the layer S2-L4. The Fe concentrations in the layers S2-L1,L2,L3 are comparable to one another for both methods, but in the layer S2-L2 the Fe concentration is significantly higher in the small size fraction. This is probably caused by the relatively high portion of strongly weathered and altered sulphide minerals in this layer, which is also seen as high Fe concentrations in the pXRF analyses. The Au concentrations ranged in all the S2 layers between 4.4 and 256 ppb.

# 4. Discussion

## 4.1. Geochemistry and environment

The XRF results show that all the four layers of Site 1 have relatively similar elemental concentrations. On the contrary, the layer S2-L4 differs from all the other layers. In this layer, the Cu, Ni, Zn and As concentrations increase considerably, and the feature is also seen in the more diverse sulphide mineral content. This plausibly indicates that the layer S2-L4 was formed, at least partially, from tailings of different ores than the layers above. However, the weathering of sulphide minerals in the surface parts could explain the high concentrations in the deeper levels.

The AAS analyses of the samples from Site 2 confirm the occurrence of certain elevated elemental concentrations and define their distribution in grain-size fractions. The Cu, Ni and As concentrations exceed the threshold values and the higher guideline values of PIMA decree (Finnish government decree 214/2007 on the assessment of soil contamination and remediation needs). Cu, Ni, and As exceed the values in all the layers, and Zn exceed those values in the layer 4. According to the PIMA decree, a soil is usually considered as contaminated if the concentration of one hazardous substance or several substances exceed the higher guideline values set for industrial, storage or transport areas or comparable areas. The higher guideline values for Cu, Ni, As and Zn are 250 ppm, 150 ppm, 100 ppm and 400 ppm, respectively. The concentrations for Cu, Ni and As exceed the higher guideline values several times in certain layers. According to Räisänen et al. (2015), the Cu and As concentrations were similarly elevated in every sample in their studies, but Ni concentrations exceeded the values in only a few samples. Generally, most metals and metalloids present in tailings are adsorbed or coprecipitated in the alkaline barrier developed in the surface layers (Smith & Huyck 1999). However, As has more complex behaviour in the wide range of pH conditions and its solubility and mobility is influenced and controlled by several factors (Salomons 1994; Marszalek & Wasik 2000). Due to the slightly acidic conditions of the sulphide-rich tailings of Rautuvaara (Räisänen et al. 2015), it could be assumed that As occurs commonly as the oxidation state $As^{5+}$ which is less mobile and toxic than $As^{3+}$ (Roussel et al. 2000; Lottermoser 2010).

In any case, the tailings ponds must be rehabilitated to prevent the release of hazardous substances into surrounding environment and the efficiency of the rehabilitation measures monitored.

Minor amounts of U-containing tailings have been deposited in the pond, but U-concentrations were found moderate at both sites. According to the XRF results the concentration in the samples does not exceed 20 ppm. The background concentrations of bedrock of the mine site are ca. 10 ppm (Northland Mines Oy 2013). However, according to Anttonen (1993), U-containing tailings from the Juomasuo deposit have been pumped in the pit located in the middle parts of the pond. Therefore, U concentrations may be locally significantly higher. In addition, it is assumed that these tailings are layered beneath the oxidized zone under reducing conditions which could indicate that U is immobile at that level (Smith & Huyck 1999).

Carbonate minerals, predominantly dolomite, are abundant in the Rautuvaara tailings. Thus, carbonates may act as a buffering agent minimizing the acid generation. According to the NP/AP-ratio (neutralization potential/acid potential: ranges <1/1 to 1/1) and pH (oxidized tailings 5.4, partly oxidized tailings 6.2) and redox measurements, the Rautuvaara tailings are potentially acid producing (Räisänen et al. 2015). The secondary mineral studies showed that the layers 1 and 2 are the most altered layers. This alteration appears as the secondary rims of Fe-oxyhydroxides, iron sulphates and Fe-oxyhydroxysulphates on the edges of the primary mineral grains. The alteration and formation of the secondary minerals were detected and particularly advanced in the layer 2 which is distinctly coloured by Fe-oxyhydroxides (see Fig. 2). The formation of the secondary minerals was discovered from the electron microprobe image (Fig. 5) as rims around the primary mineral grains. This precipitation/recrystallization could be also detected as an increase in particle size which was double in comparison to the surrounding layers. Furthermore, the iron content in the layer 2 was considerably higher than in other observed layers. Although sulphide oxidation in the surface layers was observed, it was rather slowly and moderately advancing. The 2018 rehabilitation procedure, where the tailings were covered with a synthetic membrane and glacial till, was conducted to prevent the oxidation of sulphides, the formation of AMD and to absorb water in the top layer of a substrate (Kauppila et al. 2011).

## 4.2. Mineralogy and economic potential

The versatile mineralogical character of the layer S2-L4 may be linked to the beginning of the Pahtavuoma ore enrichment in 1992. The Pahtavuoma ore was processed together with the Saattopora ore (Anttonen 1993). The coarser particle size of the layer S2-L2 indicates that the formation of secondary minerals had occurred which was also confirmed by the FE-SEM and EPMA studies. The overall downwards increase of the particle size at Site 1 could be explained by different settling times of different particle size fractions at the time of slurry pumping.

Modal mineralogy suggests that all four layers at Site 1 and the uppermost three layers at Site 2 represent the tailings formed during the same ore enrichment process where the original ore has most likely been from the Saattopora deposit. The layer S2-L4 with the deviant main mineralogy and distinctly anomalous chalcopyrite, arsenopyrite and sphalerite concentrations represent somewhat the tailings formed in the Pahtavuoma ore processing. The REE-minerals and cobaltite content of the top layers could suggest that also the tailings from the Juomasuo ore processing are mixed into those layers (Juutinen 2020).

Native gold, W-rutile and cobaltite are the most promising minerals in respect of the reprocessing of the tailings. W-rutile is abundant (100-2000 pieces of grains) in the samples obtained from Site 2, but its occurrence was almost negligible in the samples taken from Site 1. W-rutile is most likely associated with the orogenic gold ores of Saattopora and/or Juomasuo because rutile is present in their mineralogy. Apart from a single point EPMA analysis (Fig. 5), further detailed analyses of W-rutile were not performed in this study. Clark & William-Jones (2003) proposed that the ore-related metals containing rutile could be applied in ore exploration as an indicator mineral. According to them, rutile has a tendency to occur as an accessory mineral in versatile metal ore deposits showing anomalous trace element concentrations. This, together with the resistance against weathering makes rutile a potential tool in ore exploration. An association between hydrothermal ore deposits and W-rutile has been advocated by some authors (Clark & William-Jones 2003; Scott & Radford 2007; Scott et al. 2011).

The micro panning studies and GFAAS-analyses indicate that tailings could contain considerable amount of gold (15-58 pieces of Au grains; Au 4.4 – 256 ppb). Consequently, comprehensive studies on occurrence and distribution of gold are advisable. Detailed studies on whether gold occurs as pure form or as electrum (Au, Ag) were not carried out in this study. Due to the liberated form of gold, gravity separation and/or froth flotation should be considered. Additionally, cobaltite could be seen as a potential mineral to extract from tailings because of the status of Co as the critical raw material in the EU. Studies on the recovery of cobalt from sulphide tailings by acid- and bioleaching (Xie et al. 2005; Mäkinen et al. 2020) and flotation (Lutandula & Maloba 2013) has been carried out with encouraging results. More detailed characterization and reprocessing of tailings could be profitable; at least it could cover some costs resulting from the site rehabilitation.

Even though a few of the samples used in this study represent only a very small part of the tailings pond, some highly speculative calculations are presented. Assuming the Au concentrations of 0.1 ppm evenly distributed throughout the pond, the pond would contain 500 kg of Au. This calculation takes into the account mass of 5 Mt tailings (all deposited tailings except one from the Rautuvaara ore). A detailed characterization would need systematic sampling with a narrow sampling interval as, for instance, Parviainen (2009) conducted in the study concerning the characterization of the Haveri tailings. However, in the case of Rautuvaara, further actions are blocked due to the executed rehabilitation efforts.

## 4.3. Strengths and weaknesses of the applied methods

One of the main advantages of this study was the possibility to study samples in different grain size fractions. From the dry sample to slurry by adding water, decantation of the excess water and homogenization by mixing allowed the handling of small sample volumes and provided constantly repeatable results with a minimal range of variation (0-2 µm). This method also decreased (to some extent) the number of aggregates in the sample formed during drying. This method turned to be suitable both for dry samples and slurry (e.g., flotation process products) in real time, the only delay follows from time the sample requires to settling. Limitations of the method are large particle size (<0.5 mm) and materials lighter than water.

The biggest challenge of the study was, obviously, the small number of sampling sites and samples. The samples cannot represent a very large part of the area and certainly not the entire tailings pond. In respect of the heavy mineral separation, the amount of dense mineral fraction might have been too big and that is disturbing equal separation of the medium heavy material. The concentration of heavy minerals using the Knelson concentrator worked only partially for the samples from Site 1. Ilmenite, rutile, pyrite, pyrrhotite and chalcopyrite seem to be somewhat efficiently concentrated. Also, carbonates and monazite appear to be separated as heavy minerals. However, the amount of other iron oxides, for example magnetite, were not consistently concentrated. Additionally, inefficient separation with the Knelson concentrator and the heavy liquid separation occurred for the samples from Site 2 (see Supplementary Tables 2 and 3 in Electronic Appendix A). For instance, large quantities of the heavy sulphide minerals separated into the medium heavy fraction.

In addition, it is presumable that the particle size range of the tailings' material feed was too wide and should be narrowed. Mulenshi et al. (2019) and Sarala et al. (2019) have observed similar difficulties. According to them, it is possible that the bigger grains of lighter mineral displace the smaller heavy mineral grains and consequently minerals do not separate by their specific gravity. With the LST heavy liquid separation method some challenges were encountered in both projects. Sarala et al. (2019) reported that the mineral separation with LST was rough and imprecise, and sample loss was great in the Indika project. Similar issues were observed in the Biopeitto project as well.

Both preconcentration and analytical methods that were used in the study work well together. The Knelson concentrator performed quite efficiently as a preconcentration method even though some problems were encountered assumably due to the grain size range of the material feed. Also, other operation parameters than those the device manual suggested should be tested for the optimal results. Despite the semi-quantitative nature of the pXRF results, the anticipated results were obtained. Results from the portable and laboratory analyser are quite well comparable although pXRF often overestimates some elemental concentrations. In any case, the results of pXRF should always be validated by some benchmark analyser. XRD, FE-SEM and EPMA together offer great opportunities to achieve precise mineralogical results.

## 5. Conclusions

The research on the Rautuvaara tailings pond revealed that the sampled four different layers contain, at least in part, two mineralogically and geochemically different tailings materials. The three uppermost layers most likely represent the Saattopora gold-bearing sulphide ore. The fourth layer represents most likely the Pahtavuoma ore. Tailings from the Pahtavuoma ore have probably been mixed with the tailing material from Saattopora.

The Rautuvaara tailings consist of elevated concentrations of potentially harmful Cu, As, Ni and Zn. Fe-sulphide weathering occurs in the different layers of the tailings pond and further, the dissolved iron forms secondary mineral precipitates, particularly in the surface layers. The elevated concentrations of the detected metals indicate potential for future exploitation. Moreover, tentative preconcentration experiments indicate promising occurrence of gold grains in the Rautuvaara tailings. Identified W-bearing rutile grains may offer a tool as an indicator mineral in future ore exploration.

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## Supplementary Data

Electronic Appendices are available via Bulletin of Geological Society of Finland web page.

Electronic Appendix A: The detailed analytical results of the mineralogical analyses (Supplementary Tables 1–7).

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