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# Pressure leaching of copper sulphide from nickel refining: Initial experiments for valorisation

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## ABSTRACT

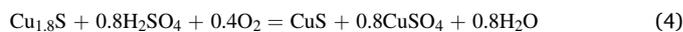
Copper sulphide rich leach residue from nickel refining is a marketable product and may be treated by smelting. In looking for another refining technology for its valorisation, the raw material was characterised and suitability of medium-temperature sulphate-based pressure leaching was tested for this purpose. The raw material contained 58% copper carried in covellite and copper oxides. Using favourable conditions, about two hours was needed for the completion of leaching. Copper extraction increased to over 99% with increasing sulphide oxidation to sulphate (92.3–99.8%) at 165 °C, and at low final acidity (20–26 g/L). In these conditions, the extraction of residual nickel was at least 99% and acid consumption 220–230 kg/t solids. The extent of sulphate formation appeared to depend on temperature, acidity and agitation. Sulphur agglomeration and scale formation occurred at low acidity between 125 and 145 °C. Iron hydrolysis during leaching was shown to remove iron from the solution to levels acceptable for electrowinning. The noble metals appeared to remain in the residue. In addition, significant portions of the impurities were released into solution. The copper dissolved could be subjected to direct recovery and the leach residue could be sold as a by-product to a precious metals refinery.

## 1. Introduction

The global production of nickel is largely based on nickel sulphide minerals (60%) with pyrometallurgical converting into matte, followed by a hydrometallurgical process with unit processes such as leaching (atmospheric and/or pressure), iron removal, solvent extraction (SX) for cobalt and nickel and final product recovery, typically by electrowinning (EW), hydrogen precipitation or crystallisation as sulphates/hydroxides/carbonates. Most refineries that process nickel matte in sulphuric acid solution selectively leach nickel over copper producing a small quantity of copper sulphide rich leach residue.

During hydrometallurgical nickel processing, one of the targets is to purify the solution to remove copper. Haavanlammi et al. (2000) have described the copper precipitation reactions in the process. They suggest that, in less acidic conditions (pH 5), nickel matte can be partially dissolved and copper precipitated as Cu<sub>2</sub>O (Eq. (1)) or as antlerite Cu<sub>3</sub>SO<sub>4</sub>(OH)<sub>4</sub> (Eq. (2)). At lower pH values (pH 2), chalcocite Cu<sub>2</sub>S contained in the nickel matte can be dissolved while digenite Cu<sub>1.8</sub>S (Eq. (3)) and further covellite CuS (Eq. (4)) can form. Under reductive conditions, e.g. during nickel pressure leaching, the dissolved copper is known to precipitate as Cu<sub>1.8</sub>S or CuS. Copper in the form of copper

sulphides is also the carrier of noble metals into further processing.



According to Fugleberg et al. (1995), the copper-rich leach residue of a nickel plant is a marketable product and suitable feed material for a copper smelter. Processing through the copper smelter route (converting – fire refining – anode casting – copper electrorefining and precious metal recovery for anode slimes) also provides efficient recovery of the noble metals contained in the residue. Mining companies with nickel refineries and copper smelters typically use the copper smelter route. The capital investments to process copper through smelting are a remarkable barrier to entry for anyone with small processing volumes. Smelters have a high capital cost and they are in favour of large capacities. Sulphur dioxide generation at the smelters requires conversion to sulphuric acid due to increasingly stringent regulations, which may have a large negative value in the economics (Everett, 1994).

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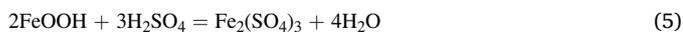
E-mail address: [pia.sinisalo@fortum.com](mailto:pia.sinisalo@fortum.com) (P. Sinisalo).

Hydrometallurgical plants are best suited for small operations.

Base metal refineries in the platinum group metal business commonly also produce copper sulphide rich leach residue and refine it further to recover copper and noble metals. These refineries typically use medium-temperature sulphate-based pressure leaching to leach copper and to leave noble metals in the residue (Plasket and Roman-chuk, 1978; Brugman and Kerfoot, 1986; Bryson et al., 2008). This technology can also be used for the treatment of chalcopyrite concentrates (Marsden et al., 2007).

Any feed originating from primary mining has a unique mineralogy with its noble minerals, base minerals and gangue minerals with varying concentrations and associations. Cobalt, copper and silver are commonly associated with nickel sulphide ores treated by nickel industry. Cobalt is recovered as side-product as well as noble metals together with copper. Only trace quantities of silver and cobalt, relatively large amounts of platinum group metals and often gold are associated with nickel-copper sulphide ores treated by platinum group metal industry. Gold is recovered together with platinum group metals while copper and nickel are side-products (Ndlovu). One technology may suit for one feed material but not necessarily to other similar feed materials without modifications. The properties of the minerals define the behavior during processing.

Base metal refineries in the platinum group metal business appear to operate pressure leaching at low acid concentration that may affect iron hydrolysis. Anderson et al. (2002) indicated that the Stillwater base metals refinery operate pressure leach process at acid concentration of 20–25 g/L and temperature of 130–135 °C. The principal reaction for iron is dissolution of hydrated ferric oxide (Eq. (5)) in the process. Magnetite, the other form of iron does not dissolve at the prevailing conditions. Hofirek and Kerfoot (1992) investigated the Rustenberg base metals refining process. They found out that at temperature of 140–145 °C ferric ion hydrolysis commenced at a moderate acid concentration of about 30 g/L. Iron may precipitate as jarosite (Eq. (6)) or as hematite (Eq. (7)).



The current study explores for a suitable hydrometallurgical refining technology for the valorisation of copper residue from a nickel refinery. The main objective was to leach copper and any remaining nickel while concentrating precious metals in the solids. The material was characterised and a potential method i.e. medium-temperature sulphate based pressure leaching was tested for the material. In particular, the effects of agitation, temperature and iron hydrolysis were investigated. Agitation may limit leaching kinetics. Temperature has an effect on sulphur deportment that may interfere with leaching. Iron hydrolysis may remove iron from solution to required level already during leaching and hence serve an outlet for iron in a process.

## 2. Experimental

### 2.1. Raw material

The raw material investigated was the copper sulphide leach residue of an industrial nickel refinery. The sample used was dried at 60 °C, homogenised by rolling and divided using a rotary sample divider. According to the sample provider, the material typically had a moisture content of 20%. The sample contained 12% moisture measured with a moisture meter and the weight loss during drying was 7%. This suggests that the sample investigated may have partly oxidised during storage and may have oxidised further during drying. The phenomenon is typical for this type of intermediate residue of nickel production. The divided sub-samples were stored in a fridge to hinder further oxidation.

The density of the sample was 4.3 kg/L according to gas pycnometer determination. The sample was measured to have P80 of 40 µm by laser particle size analysis (Malvern Mastersizer 3000, wet conditions).

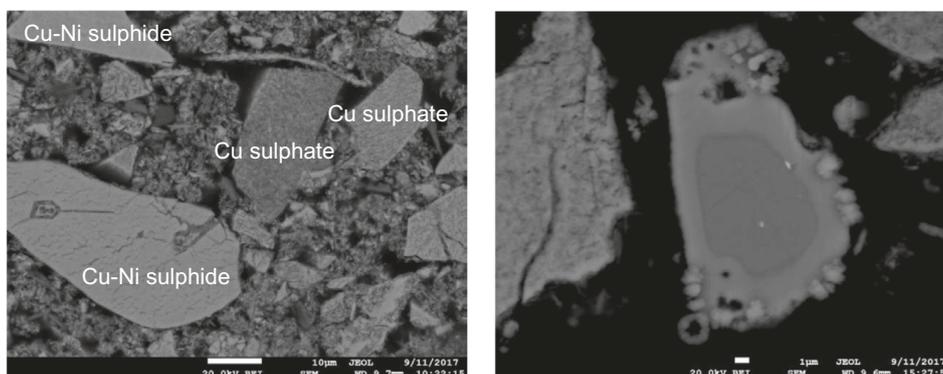
The sample was poorly crystallised. X-ray diffraction analysis (Bruker D8 Discover A25 diffractometer) showed the presence of covellite (CuS), antlerite (Cu<sub>3</sub>(SO<sub>4</sub>)(OH)<sub>4</sub>), brochantite (Cu<sub>4</sub>(SO<sub>4</sub>)(OH)<sub>6</sub>) and chalcantite (CuSO<sub>4</sub>·5H<sub>2</sub>O). A field emission scanning electron microscope equipped with an energy dispersive spectrometer analyser (FE-SEM: JEOL JSM 7100F Schottky) also indicated the presence of covellite and copper sulphates. Most of the phases identified from the sample were copper-nickel sulphides with varying copper and nickel content as well as copper sulphates (Fig. 1). In addition, iron-copper sulphates, copper-nickel-iron-cobalt sulphides and nickel-iron-cobalt sulphides were present. Also, some lead-rich phases were identified. The analyses were conducted at GTK Research laboratory, Espoo.

The desired element content of the sample was analysed after four-acid leaching by inductively coupled plasma, combustion analysis and combined fire assay atomic absorption techniques (Labtium). The copper content was also analysed by using sequential phase analyses (Outotec Research Center, Pori). The copper phase assay consisted of sequential copper analyses after the residue had been leached in four different media: water, sulphuric acid, cyanide and nitric acid. The copper sulphates (water), copper oxides and silicates (H<sub>2</sub>SO<sub>4</sub>), secondary copper sulphides such as covellite (cyanide) and primary copper sulphides such as chalcopyrite and metallic copper (HNO<sub>3</sub>) can be chemically distinguished by this method. The sample was shown to contain 58% copper together with 3% nickel, 23% sulphur and 0.1% noble metals (Table 1). It also contained 0.2% selenium and other impurities. 61% of the copper was found in covellite, 36% in copper oxides and basic copper phases with minor amounts present as copper sulphate and primary copper sulphide phases.

### 2.2. Leaching test series

The factors under investigation in the test work were the temperature and iron concentration of the feed solution in addition to agitation. The sulphuric acid concentration was varied in the experiments (initial from 43 to 90 g/L) – the target being 20 g/L after the copper had been leached. However, it was found that the actual value of final acidity varied due to the behaviour of sulphur (and iron) for the different test conditions examined. An oxygen overpressure of 7 bar was chosen, as according to Fleming et al. (2008) this has a minimal effect on the rate of oxidation of the sulphides above about 5.2 bar. Also, 7 bar is standard in the gold industry where sulphur is oxidised to sulphate. Enough long leaching time was estimated for the tests for the completion of copper leaching, and was revised during the test work between three and four hours. Table 2 presents the conditions of the experiments performed and the final acidity of the solution.

Pressure leaching tests were conducted in a titanium autoclave with an effective volume of 0.8 L (Büchi ecoclave 075). Synthetic solution and copper sulphide leach residue were added to the autoclave. Synthetic solution was prepared from reagent-grade sulphates. The agitation was switched on and the pulp heated after closing the reactor. The temperature was controlled by electric heating elements located around the reactor shell. The pulp reached 145 °C typically within 30 min. Oxygen was allowed into the reactor once the desired temperature had been reached. This time was considered as the start of the leaching process. The pulp reached the desired pressure typically in less than a minute. Pressure was maintained at the desired value by manual control of the oxygen feed rate, while a small purge stream which evaporated a minimal amount of water from the reactor was used. The purge stream was applied after the first test, as the agitation power was decreased. Samples were taken manually with a sampling system having a 7 µm filter at the end of the sampling tube. Once the sample was taken, it was immediately filtered with a syringe filter to remove any possible particles from the sample. The reactor was cooled down by passing cooling



**Fig. 1.** Backscatter electron micrographs of the copper sulphide leach residue (FE-SEM: JEOL JSM 7100F Schottky). Copper sulphides and sulphates (left). Ni-Fe-Co sulphide (core) covered by Cu-Fe-Ni-Co sulphide (right). The two bright inclusions are lead sulphide (right).

**Table 1**

Chemical composition of the investigated copper sulphide leach residue.

Cu	4-acid leach	57.6	%	S	Combustion	22.5	%
Ni	4-acid leach	3.2	%	Si	Peroxide fusion	0.4	%
Fe	4-acid leach	1.5	%				
Co	4-acid leach	0.2	%	Se	4-acid leach	1980	ppm
				As	4-acid leach	280	ppm
Cu	P1	1.4	%	Te	4-acid leach	180	ppm
	P2	20.1	%	Sb	4-acid leach	78	ppm
	P3	33.8	%	Bi	4-acid leach	62	ppm
	P4	0.5	%				
	Sum	55.8	%	NB	Fire assay	1000	ppm
					of which Ag	920	ppm

P1 = H<sub>2</sub>O-soluble, P2 = acid-soluble, P3 = cyanide dissolution, P4 = nitric acid dissolution, NB = noble metals.

**Table 2**

Test conditions. The solids and copper concentrations were 90 g/L and 15 g/L initially. Oxygen overpressure was 7 bar. The target for acid in discharge was 20 g/L after copper had been leached.

Test no	P/V kW/m <sup>3</sup>	T °C	Fe(III) <sup>1</sup> g/L	Ni <sup>1</sup> g/L	Purge stream	Time <sup>2</sup> h	Acid <sup>1</sup> g/L	Acid <sup>3</sup> g/L
1	5.6	150	–	15	No	4	90	59
2	2.4	165	5	5	Yes	3	43	26
3	2.4	165	–	5	Yes	3	43	20
4	2.4	145	1	5	Yes	3	56	33
5	0.5	145	1	5	Yes	3	56	36
6	2.4	125	–	5	Yes	4	43	16
7	13	145	1	5	Yes	3	56	26

1 = Feed solution, 2 = leaching time after desired T was reached, 3 = acid in discharge.

water through the reactor shell after leaching. The warm pulp was removed from the reactor for instant filtration and solids washing. Solids were dried at 60 °C. The chemical analysis of samples was conducted by inductively coupled plasma spectrometer techniques. The solid samples were analysed after aqua regia digestion. Peroxide fusion was used as the dissolution method for sulphur. Noble metals were analysed by the combined fire assay atomic absorption technique. Acid was determined by pH titration to fixed end point of 3.2 to avoid the dissolved metals precipitation. This titration method makes a small error, as acid concentration is less than 0.5 g/L at pH 3.2. Element extractions were calculated based on feed analysis and discharge solids analysis.

### 3. Results and discussion

#### 3.1. The effect of agitation and residence time

The first factor investigated was agitation. To provide process conditions and basic kinetic data for the process development, chemical

batch tests should be carried out in conditions where the reaction rate is not limited by the mixing intensity (Latva-Kokko et al., 2016) or availability of reagents. Tests were done using a pitched blade turbine with three blades (Fig. 2). The highest agitation speed of the equipment was 1000 rpm although the first test was done at 1300 rpm. A radial flow turbine with six blades (Fig. 2) and a new shaft were ordered with which the last test was performed at 1200 rpm. The power intake in each test was estimated using Eq. (1).

$$P = N_p \rho n^3 D^5 \quad (1)$$

where P is agitation power (W), N<sub>p</sub> is power number (-), ρ is pulp density (kg/m<sup>3</sup>), n is agitation speed (1/s) and D is impeller diameter (m).

The power number for a pitched blade turbine (3-blade, 45°, blade widths 1/5 of the diameter) is 1.19 (Chemical Processing, 2013) and for a Rushton turbine with six blades 6.0 (Post Mixing, 2013). These were used as the estimates of the power numbers for the turbines used. A pulp density of 1100 kg/m<sup>3</sup> was estimated. The same density estimate was used when calculated from Tiihonen et al. (2013) that up to 11–19 kW/m<sup>3</sup> power intake was used in their mass transfer tests that were carried out in a 4-L autoclave. The power intake of an industrial-scale autoclave operating under similar agitator duty may be up to 2.3–2.9 kW/m<sup>3</sup> in a limited number of cases (Nicolle et al., 2009).

The selection of agitation intensity was shown to have a significant effect on the copper extraction rate. It took an hour for secondary copper sulphides to begin to leach at a power intake of 2.4 kW/m<sup>3</sup> while the same was achieved in 0.5 h at power intakes of 5.6 and 13 kW/m<sup>3</sup> (Fig. 3). Fig. 3 also shows that at time instant 0 h i.e. during the autoclave heating period, approx. 30% of copper had already dissolved. This indicates that acid-soluble copper leached with fast kinetics i.e. before the tests were considered to have started. The achieved extraction of copper in test 7 was lower than in tests 4 and 1, due to sulphur agglomeration.

The secondary copper sulphides were shown to leach faster at a higher power intake per unit volume due to the higher mass transfer of



**Fig. 2.** Impellers used in the test work. A pitched blade turbine, diameter 50 mm (left). A radial flow turbine, diameter 45 mm (right).

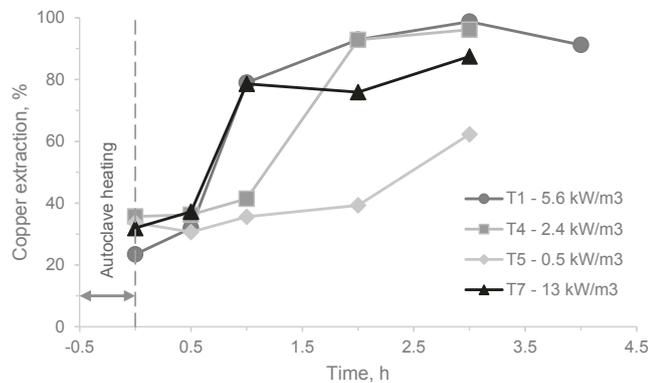


Fig. 3. Effect of agitation power on leaching of copper in tests T1, T4, T5 and T7.

oxygen to the liquid phase in the tests. Results from Latva-Kokko et al. (2016) show that agitation power per unit volume is the dominant factor for gas feed capacity below the flooding point (direct relationship) while also the impeller design has some impact. In other words, the utilisation of a certain amount of gas is generally higher when the agitation power per unit volume is increased. This is due to better gas dispersion.

A well-agitated solids suspension is typically achieved at a power intake of less than 1 kW/m<sup>3</sup> for raw material similar to that used in the current study. However, this is seldom high enough, especially in pressure leaching where the oxygen feed rates are typically high. The rate-limiting step in hydrometallurgical operations due to poor oxygen solubility and relatively high oxygen demand is often the mass transfer of oxygen from gas to liquid (Latva-Kokko et al., 2016).

The results also suggest that approximately two hours was needed for the completion of copper leaching using favourable conditions (Fig. 3). At this time, copper extraction had reached the plateau in test 1. Other studies using oxygen for similar feed material (Plasket and Romanchuk, 1978; Brugman and Kerfoot, 1986) suggest similar residence times. In the current study, each leaching test lasted for at least three hours, which was long enough for the copper to leach, apart from test 5 for which the agitation power was lowest.

### 3.2. The effect of temperature and sulphur deportment

Other factors investigated in the leaching test series were temperature and iron hydrolysis. Moderate pressure leaching temperatures were investigated (125–165 °C), similar to those used in several plant operations (Hofirek and Kerfoot, 1992; Plasket and Romanchuk, 1978; Anderson et al., 2002; Brugman and Kerfoot, 1986). Above the melting point of sulphur i.e. 120 °C, sulphur tends to wet sulphide mineral surfaces, which can interfere with mineral oxidation (Marsden et al., 2007). Sulphur is generally considered to oxidise to sulphate fully above 200 °C within the time frame of leaching operations. Metallurgical plants typically contain internal recycles that accumulate impurities within the circuits (Hofirek and Halton, 1990). In many cases, iron hydrolysis has an important role in iron removal from solution and may have a role in the collection of non-metallic impurities (Hofirek and Kerfoot, 1992). Iron hydrolysis was investigated by varying the iron concentration of the feed solution (0–5 g/L). This is a typical iron recycle concentration from electrowinning (Robinson et al., 2013).

Fig. 4 presents the copper extraction and the extent of sulphate formation in experiments T2–T4 and T6 where the mixing intensity was the same in all tests, i.e. 2.4 kW/m<sup>3</sup>. Also, T7 is presented (with a high mixing intensity of 13 kW/m<sup>3</sup>). It can be seen that copper extraction increased with increasing sulphate formation in these tests (Fig. 4). Tests performed at high temperature (tests 2 and 3 at 165 °C) yielded the highest copper extractions, over 99.8%, and over 90% sulphate formation. Nickel extractions (99.5% in test 3 and 98.7% in test 2) were also

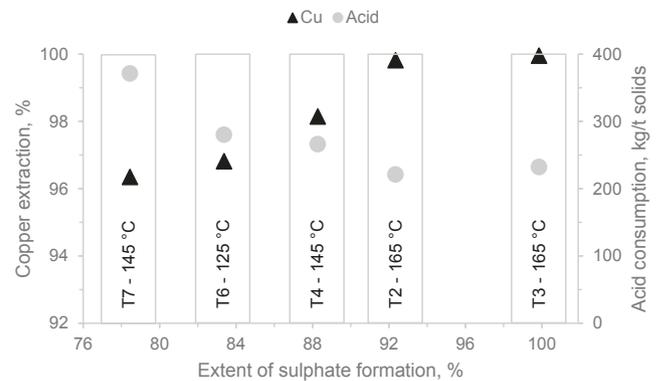
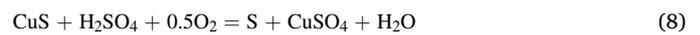


Fig. 4. Relationship between copper extraction (based on solid analysis), extent of sulphate formation, temperature and acid consumption in tests T2–T4 and T6–T7.

the highest in these tests. The extent of sulphate formation (over 78%) and mineral leaching (over 96% copper extraction) was less in the other tests. The presence of elemental sulphur hindered sulphide mineral leaching. Agglomerates were observed in residues from the tests yielding the two lowest extractions (T6–T7), while scale formation was observed on the filter of the sampling tube in test 4 (Fig. 5). The acid consumption decreased with increased sulphate formation in the tests (Fig. 4). Sulphide oxidation to elemental sulphur consumes acid (Eq. (8)) and sulphate formation produces acid (Eq. (9)). Increased iron precipitation due to the iron addition in test 2 also produced some acid (Eqs. (6) and (7)). It can be seen that at high temperature (165 °C) the acid consumption was approx. 230 kg/t solids, whereas at 125–145 °C the consumption varied between 270 and 370 kg/t solids.



The extent of sulphate formation was shown to depend on temperature. The relationship was direct as the results from tests 6, 4 and 3 show in Fig. 4. An increase in temperature is well known to increase sulphate formation as well as to lower the elemental sulphur content of the residue. Elemental sulphur interfered with mineral oxidation in the tests between 125 and 145 °C. In addition, the different agitation intensity in test 7 (increased power intake and radial flow pattern) in comparison with test 4, where both tests were at 145 °C, apparently caused the formation of relatively large agglomerates (Fig. 5, T7), which interfered with mineral oxidation (Fig. 4). The agitation pattern used was axial, apart from in test 7.

Relatively high acidity may hinder sulphate formation. At about 150 °C, copper extraction was approx. 98% in tests 1 and 4, whereas the extent of sulphate formation was 78% in test 1 and 88% in test 4. Test 1 contained twice as much acid than test 4 (discharge acid 59 g/L compared to 33 g/L). Copper extraction was over 99% in tests 2 and 3 whereas extent of sulphate formation was 92% in test 2 and over 99% in test 3, with a temperature of 165 °C. Test 2 had slightly higher acidity during leaching (discharge acid 26 g/L compared to 20 g/L). Marsden et al. (2007) concluded that sulphide conversion to elemental sulphur depended on the temperature and acidity during pressure leaching of chalcopyrite. This also agrees with the results described here.

Challenges related to sulphur wetting also include the physical transfer of the pulp due to the presence of sulphur agglomerates (Marsden et al., 2007). The addition of sulphur dispersants such as lignosulphonates is well known and these can be used to reduce sulphur wetting. Dreisinger (2006) describes some moderate temperature pressure leaching processes such as that of Dynatec in which sulphur dispersants are used for chalcopyrite leaching. However, no dispersants were applied in this study.

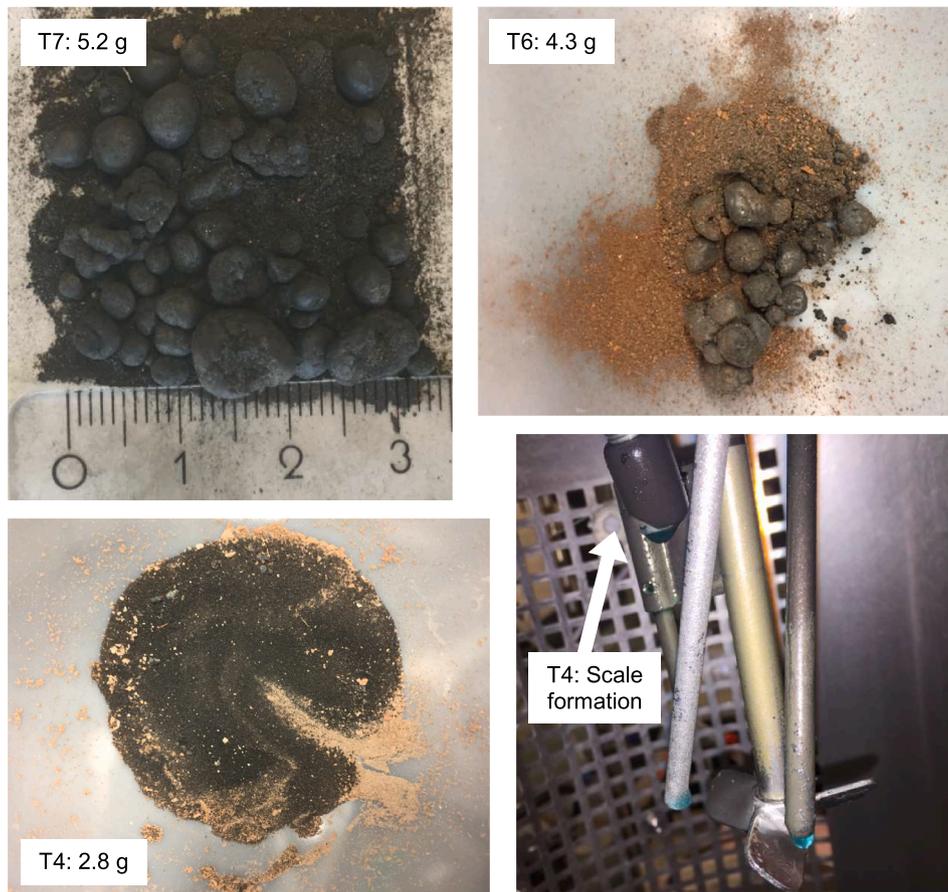


Fig. 5. Residues (T7, T6, T4) and observed scale formation on sampling tube filter. Black agglomerates cemented by sulphur. As the amount of agglomerates decreases, the brown to pale powder will be indicative of a combination of elemental sulphur, unleached trace minerals and small amounts of iron precipitates.

### 3.3. Behaviour of iron and other impurities

The amount of iron in solution increased by about 1 g/L during leaching due to iron originating from the raw material apart from test 2 (with 5 g/L initial iron concentration in the head solution). In test 2, the iron concentration in the discharge solution was found to be the same as in the feed solution, with a discharge acid concentration of 26 g/L. Thus, the 5 g/L addition of iron in test 2 favoured net iron hydrolysis. Iron addition of 1 g/L in some of the tests (T4, T5, T7) had an insignificant effect on net iron hydrolysis. This was probably due to the relatively high discharge acidity in the tests, as lower acid concentration is known to favour iron hydrolysis (Marsden et al., 2007).

Iron precipitation was found to change the residue composition and produce a larger residue mass, see Fig. 6. Experiments T2 and T3

resulted in the highest copper extraction (Fig. 4), although the mass loss varied. T2 with 5 g/L of iron in the head solution had a mass loss of 85% whereas in T3 with no additional iron the mass loss was higher i.e. 91%. Iron precipitated in T2 while iron extraction was 85% in T3. Iron precipitated probably as iron compounds other than hematite in test 2 based on the residue colour. The presence of iron as only jarosite in the residue was not supported by chemical analyses. Typically, mixtures of goethite, hematite and jarosite can produce.

Significant portions of impurities were released into solution. Discharge concentration levels were generally 200 mg/L cobalt, 130 mg/L selenium, 20 mg/L tellurium, 20 mg/L arsenic, 10 mg/L antimony and 3 mg/L bismuth. On the whole, nearly all of the cobalt was leached and the extraction of the other impurities were relatively high. It seems that copper, cobalt and selenium started to leach at the same time and

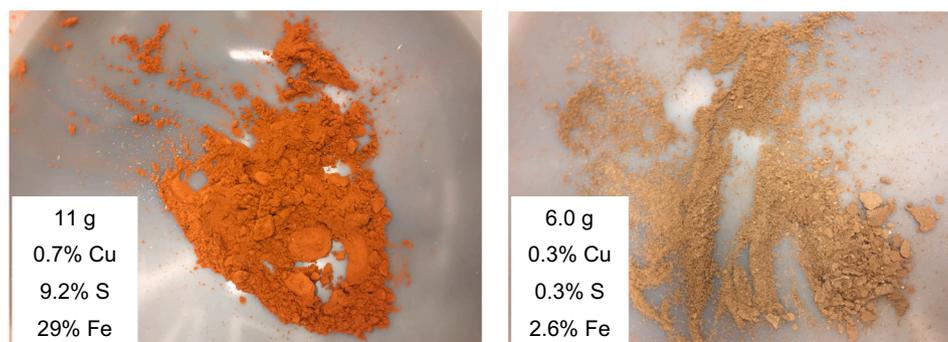


Fig. 6. Residues from T2 (left) with 5 g/L of iron in the head solution and T3 (right) with 0 g/L of iron in the head solution.

their leaching behaviour was very similar. This suggest that selenium was hosted in the same minerals as copper and cobalt.

The discharge residues were calculated to contain less than 3% noble metals based on mass loss. This residue was analysed to contain 1.5% noble metals (1.6% based on mass loss) in test 6 and 1.0% noble metals (1.3% based on mass loss) in test 7. In the investigated conditions, some silver may have leached while the other noble metals appeared to remain in the residue. According to [Crundwell et al. \(2011\)](#), the precious metals refinery in Acton, England processes feed material with less than 15% noble metals, suggesting that the leach residue could be further sold as a by-product whereas the copper dissolved could be subjected to direct recovery. However, the feeds to precious metals refineries contain more typically between 50 and 70% noble metals.

#### 4. Conclusions

In the current study, the medium-temperature pressure leaching for valorisation of copper sulphide leach residue from a nickel refinery was investigated. The residue contained 58% copper, which was carried by covellite (CuS), antlerite (Cu<sub>3</sub>(SO<sub>4</sub>)(OH)<sub>4</sub>), brochantite (Cu<sub>4</sub>(SO<sub>4</sub>)(OH)<sub>6</sub>) and chalcantite (CuSO<sub>4</sub>·5H<sub>2</sub>O). Copper extraction was constantly over 96% and sulphide conversion to sulphate at least 78%. About two hours was needed for the completion of leaching using favourable conditions. It seems that lower acidity and higher iron concentration (test 2 with 26 g/L discharge acid and 5 g/L iron in head solution) favoured net iron hydrolysis which can remove iron from the solution to levels acceptable for electrowinning. Noble metals appeared to remain in the residue during leaching.

The maximum extraction of copper coincided with the maximum conversion of sulphides to sulphate. Tests performed at high temperature (tests 2 and 3 at 165 °C) and lower acidity (discharge acid 20–26 g/L) where the mixing intensity was 2.4 kW/m<sup>3</sup> (axial flow pattern) yielded the highest copper and nickel extractions, both over 99%, and over 90% sulphate formation. The extent of sulphate formation appeared to depend on temperature (direct relationship), acidity (high acidity may hinder sulphate formation) and agitation (increased power intake and radial flow pattern apparently caused formation of relatively large sulphur agglomerates, which interfered with mineral oxidation). The presence of elemental sulphur hindered sulphide mineral leaching between temperatures of 125 and 150 °C.

The results show that medium-temperature pressure leaching is a potential method for valorisation of copper residue from a nickel refinery. The copper dissolved could be subjected to direct recovery and the leach residue could be further sold as a by-product to a precious metals refinery. The findings may interest those who seek alternative options to smelting to generate value for copper-containing nickel refinery side-product. To develop and evaluate the valorisation process in overall, solution purification and copper recovery stages should also be defined and evaluated. To realize optimal full-scale solution from a design perspective, the identified conditions must be evaluated in pilot-scale.

#### CRedit authorship contribution statement

**Pia Sinisalo:** Investigation, Conceptualisation, Validation, Formal analysis, Writing - original draft, Writing - review & editing. **Shila Jafari:** Investigation. **Mari Lundström:** Funding acquisition, Project administration, Resources, Writing - review & editing.

#### Declaration of Competing Interest

The authors declare that they have no known competing financial

interests or personal relationships that could have appeared to influence the work reported in this paper.

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