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Material Value Chains

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Characterization and recovery of dusts from mechanical treatment of WEEE



Solution Architect for Global
Bioeconomy & Cleantech Opportunities



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**Characterization and recovery of dusts
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Name of the report: Characterization and recovery of dusts from mechanical treatment of WEEE

Key words: Dust, WEEE, flotation, leaching, metals

Summary

Current recycling of WEEE composes of several stages in which materials are separated from each other to produce feeds for raw materials industry. During the mechanical treatment, fractions that are not further recovered are also produced. One of these fractions are the valuable dusts from different locations of the mechanical treatment chain.

The aim of this study was:

- Characterize dusts from mechanical treatment to identify characteristics that can be taken into account in the further treatment.
- Develop and apply possible treatment methods to the dusts in order to recover valuable materials which are today lost

The study revealed heterogenic and complex nature of dusts which vary in composition and particle size depending on the origin of dust in the process. By combining sieving and flotation, metals could be enriched with an enrichment ratio between 1.1 and 1.25. Highest enrichment ratio was detected with gold. Around 60 % of TOC was separated by primary sieving to an overflow fraction. Chemical leaching revealed that majority of heavy metals can be removed from dust with rather strong HCl or H₂SO₄, and elevated temperatures. However, especially lead is easily left in the solid residue. The gold was dissolved with Aqua regia. With gold, analysis were problematic, as controversial results were obtained when determining gold from different analytical and calculative methods. According to residual solid based calculation, Au dissolution was ~90%, requiring no acid leaching as pretreatment. However, feed solid based analytics indicated that strong sulfuric acid leaching was required as pretreatment, and still Au dissolution was only 60%.



Helsinki, 22 June 2017





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1 Background

Current recycling of WEEE composes of several stages in which materials are separated from each other to produce feeds for raw materials industry. Main objective of the mechanical material separation is to separate plastics from metals and further on the metals to own main metal concentrates such as copper and iron concentrates. During this mechanical treatment fractions that are not further recovered are also produced. One of these fractions are the dusts from different locations of the mechanical treatment chain. Usually these dusts origin from the crusher but they can migrate to other parts of the treatment chain where they are collected for instance with cyclones. These dusts has been identified in the international literature to contain both valuables and harmful elements (Bachér et al. 2015; Bachér and Kaartinen, 2017; Chancerel et al. 2009; van Schaik and Reuter, 2014)

The aim of this study was to characterize dusts from mechanical treatment to identify characteristics that can be taken into account in the further treatment. In addition, possible treatment methods were developed and applied to the dusts in order to recover valuable materials which are today lost. Beside metals recovery, environmental aspects were taken into account but acted not as a driving force.

2 Materials and methods

2.1 Sample

For the study three dust samples (Dust 1, Dust 2, Dust 3) was received from Kuusakoski's large scale treatment trials in which low grade printed circuit boards from cathode ray tube displays and other monitors were processed. The dust samples were from three different stages of the process and presented together roughly 1.3 w% of the total amount processed material. From this proportion Dust 1 present roughly 65 w% of the dust sample whereas for Dust 2 and the 3 the proportions are 30 w% and 5 w% respectively.



Figure 1. Primary dust sample in this study. From left to right: Dust 1, Dust 2, Dust 3.

2.2 Characterization

Particle size distribution was determined by sieving 500-720 g of sample material with 10 mm, 4.0 mm, 1.0 mm, 0.50 mm, 0.355 mm, 0.25 mm and





0.125 mm sieves for 20 minutes. The analysis was performed with Retch AS300 analytical sieve shaker.

Elemental composition of the sample was determined by using Axios mAX 3 kV X-ray spectrometer and semi-quantitative fundamental parameters program (RRFPO). The method is applicable for fluorine and elements heavier than fluorine, and a typical detection limit is approximately 0.01 w-%.

Gold content was determined with Pb-Fire Assay method combined with ICP-OES. The pulverized sample is weighed and mixed with a fluxing agent. The sample is then heated in a furnace where it fuses and separates from the collector material 'button', which contains the precious minerals. Once the button is separated from the gangue, the precious metals are extracted from the collector through a process called cupellation. Once the button has cooled, it is separated from the slag and cupelled. When lead is used as a collector, the lead oxidizes and is absorbed into the cupel leaving a precious metal bead. The bead is then dissolved in aqua regia for analysis. The gold is analyzed from the aqueous sample with inductively coupled plasma optical emission spectrometry (ICP-OES)

Total organic carbon (TOC) was determined according to standard SFS-EN 13137 using an element analyzer. In the method, solid sample is combusted in an oxygen flow (1400 °C), and carbon dioxide formed in combustion is determined on an IR-detector. Before combustion, the sample is acidified with phosphoric acid to remove inorganic carbon.

2.3 Treatment

The treatment of dust was carried out on a composed sample "Dust Mix" (Figure 2) from all three samples received from Kuusakoski Oy. Prior to the treatment, Dust 1 and Dust 2 was sieved with 0.355 mm sieve. The underflows from sieving were mixed together with Dust 3 based on the shares of generation in Kuusakoski's process. In overall, this "Dust Mix" sample present roughly 67 % of the total dust amount. Overflows from sieving (~23 %) was stored. This fraction could potentially be utilized in energy recovery.





Figure 2. "Dust Mix" sample for the treatment studies.

Dust Mix sample underwent a series of flotation and leaching experiments. In flotation experiments, the target was to enrich metals to one fraction while organics and other harmful elements would be separated to the other fraction. The flotation experiment composed of three trials in which different configurations in a two-step flotation circuit was examined. Leaching experiments composed of several leaching steps to selectively leach and separate metals from the sample. Dust Mix was used as the feed for the leaching experiment. As a result of the treatment, metals could be separated from the dust leading possibly also to improvements in environmental properties of the material together with organics removal by flotation and sieving.

In hydrometallurgical treatment the main goal was to understand how different leaching chemicals (nitric-, hydrochloric-, sulfuric acid) and their strength, as well as other leaching parameters (temperature, time) affected to the dust and its elements. Dust mix was found to be very challenging material, due to the presence of TOC, which causes usually increased acid consumption and decreased yields for valuable elements, especially for gold. Iron and aluminum, major elements of dust mix, are also known to consume excess leaching chemicals. Therefore, possibilities for their removal prior to gold leaching was studied.



3 Results and discussion

3.1 Characterization

In order to develop possible treatment methods for dust, the chemical composition and physical properties (particle size) need to be known. This chapter presents the characterization results.

3.1.1 Particle size

The particle size distribution of all three dust samples is presented in Figure 3.

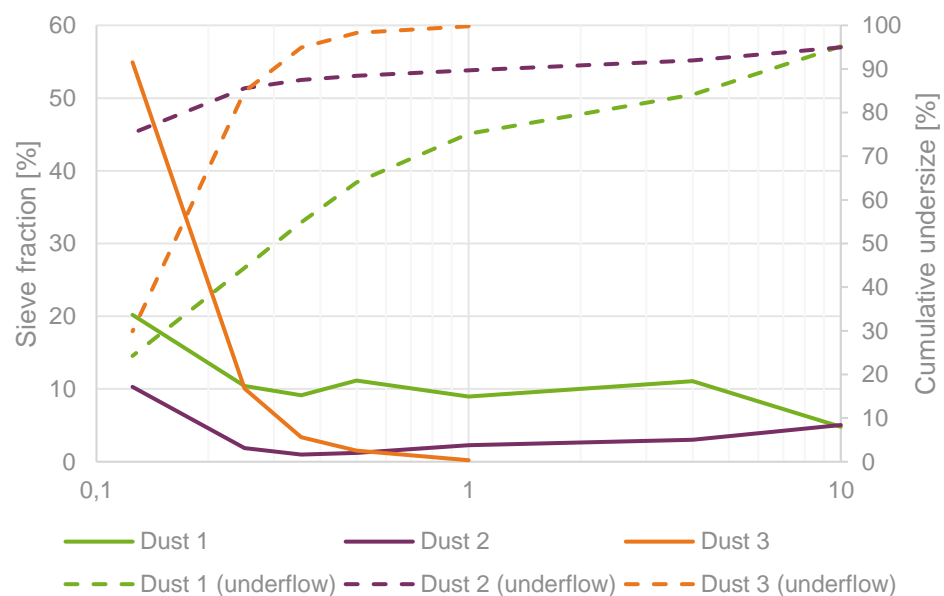


Figure 3. Particle size distribution of dust, size in mm.

The particle size distribution varied between all three dust samples (Figure 3). The d80 value, representing the cut size in which particles have 80 % chance of passing the sieve was 2.0 mm, 0.19 mm and 0.24 mm for Dust 1, 2 and 3 respectively. From Figure 3 it can be noticed that Dust 1 has the broadest distribution composing more of larger particles than for the other two dust samples. The d80 value is on similar range for both Dust 2 and 3 samples. However, when examining the distributions, it can be seen that a major share (~75 W%) of Dust 2 is composed of particles with the size of below 125 μm where as a significant share (~55 w%) of Dust 3 particles have the particle size 125-250 μm .

3.1.2 Chemical composition

Primary dust samples

Chemical composition analysis in which all generated sieve fractions were analysed revealed differences between the dusts in terms of content and element distribution.



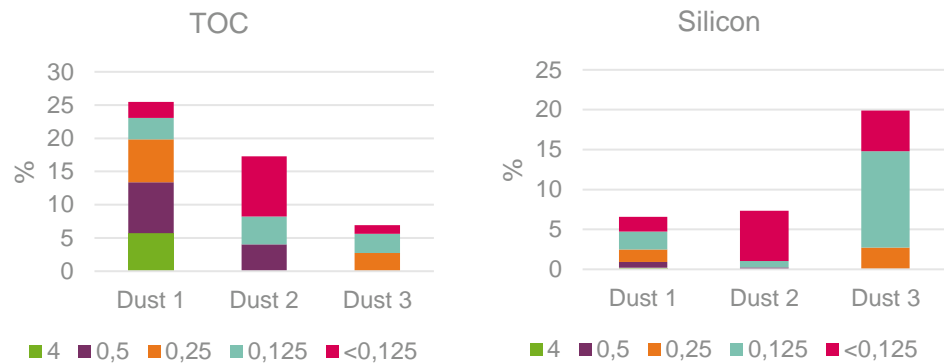


Figure 4. TOC and silicon content and distribution in dust samples.

Non-metallic components such as TOC and silicon were the most common component/element in the dusts (Figure 4). TOC content varied between 7 and 25 % depending on the dust. As for silicon the content varied between 6 and 20 %. When comparing TOC and silicon concentrations, it can be noticed that when TOC concentration is high silicon content is low and vice versa. The dust origin location in the mechanical treatment chain as well as the generation mechanism most probably causes this difference. As for the distribution of these components, it can be seen that TOC distributes more evenly between the size fractions than silicon which concentrates to the finer particles.

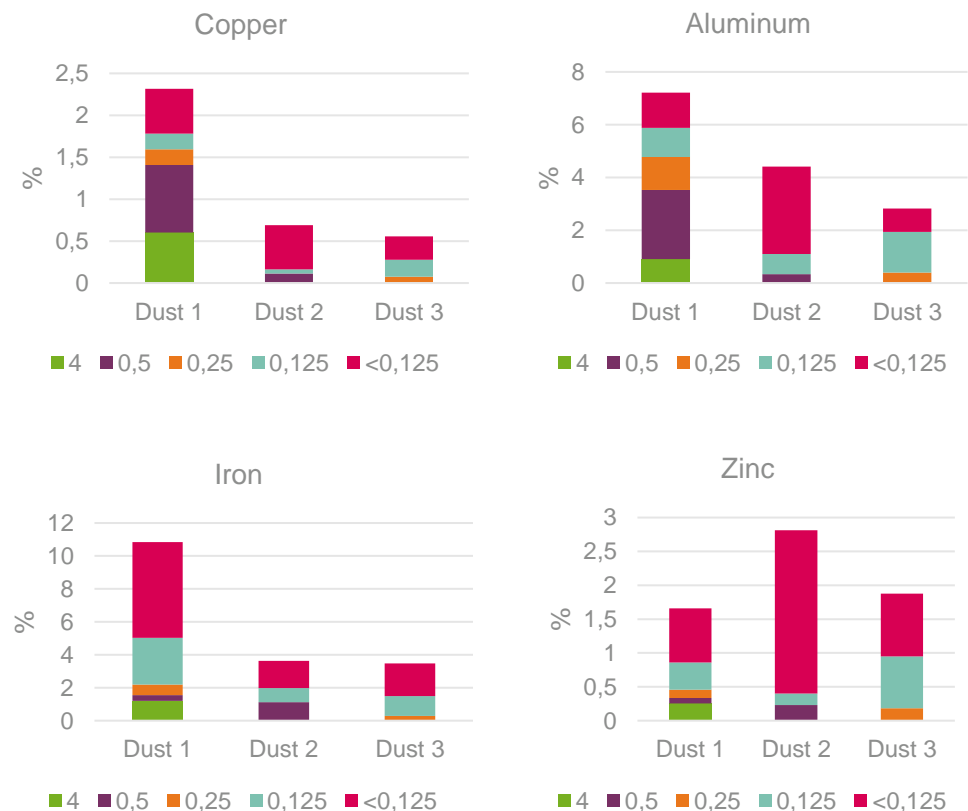


Figure 5. Copper, aluminum, iron and zinc content and distribution in dust samples.





Base metal content varied from roughly 0.5 % (copper) up to 10 % (iron) measured with semi quantitative XRF (Figure 5). Copper, aluminum and iron content in Dust 1 were remarkable higher than in the other dusts. As for other base metals such as nickel, zinc and tin the difference were not so significant. Base metals concentrated mostly in all dusts to the finer particles with the exception of copper and aluminum which distributed rather evenly between all size fractions in Dust 1.

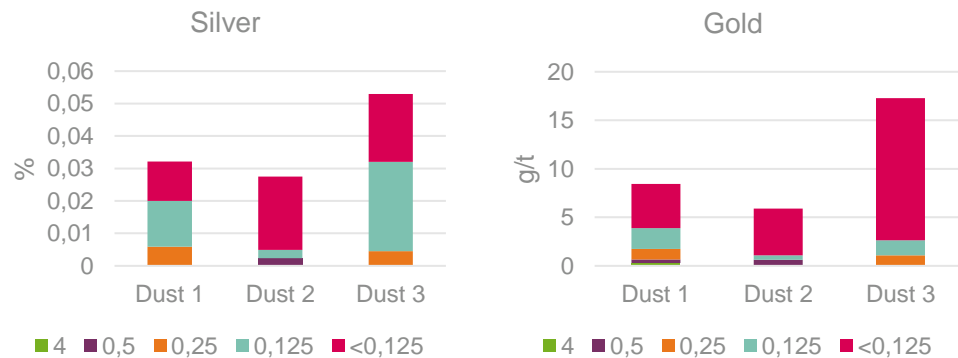


Figure 6. Silver and gold content and distribution in dust samples.

For precious metals higher concentrations could be seen for Dust 3 (Figure 6). This trend could be identified especially for gold. The origin location and generation mechanism for Dust 3 in the treatment chain produces dusts with higher precious metal content. The precious metals seem to concentrate to the finer particles which makes them especially challenging to recover since smaller particles tend to lose more easily than larger particles.

Dust mix sample

An overall element composition of the Dust mix sample, analyzed with ICP-MS/OES, is presented in Figure 7 and Figure 8.

Dust Mix material content (1/2)

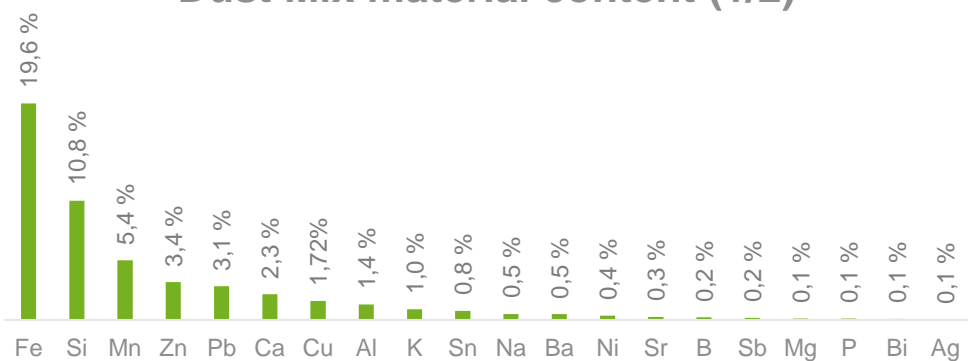


Figure 7. Dust Mix material content (1/2).





Dust Mix material content (2/2)

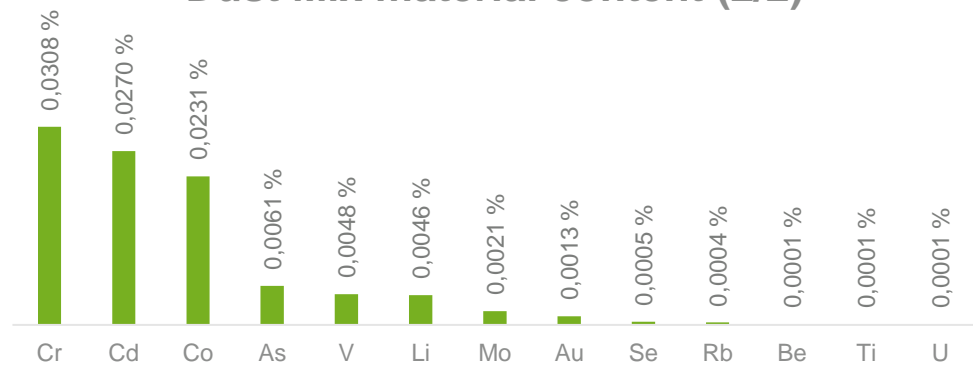


Figure 8. Dust Mix material content (2/2)

It can be noticed that the ICP analysis resulted in much higher iron content compared to XRF analysis which was carried out on the three dust samples presented in previous chapters. Differences were identified also for other elements such as aluminum. However, it was seen valuable to make the XRF analysis for the primary dust samples to identify/screen the elements in the samples whereas for leaching experiment the ICP analysis was seen more suitable.

3.2 Treatment

The aim of the dust experiments was to study potential treatment methods to recover metals from dusts and separate possible harmful substances for safe disposal. In this chapter, both the results from flotation and leaching experiments are presented.

3.2.1 Flotation

The flotation experiments composed of three trials with different configurations. First flotation trial composed of two step flotation in which the second flotation step was carried on the froth fraction from the first step. The second flotation trial composed of the same flotation steps with similar running parameters than the first flotation trial except a fines removal (<45 µm) was carried out prior the flotation. In the third flotation trial, the flotation was carried out on two step concentrate flotation with reagent addition.

In Table 1. the cumulative mass pull of flotation experiments has been presented.





Table 1. Cumulative mass pull separated with flotation from Dust Mix to froth fraction. Flotation 2 +45 µm stands for the combined sample of concentrate from flotation and fines (<45 µm) from sieving.

Cumulative mass pull (w%)	
Flotation 1	30.8
Flotation 2	27.3
Flotation 2 + 45 µm	20.5
Flotation 3	31.3

Significant differences in the mass pull between three flotation trials did not occur. Slightly lower mass pull was identified for Flotation 2 trials since the fines has already been separated prior to the flotation and therefore less particles suitable for flotation exists in this trial. In addition, Flotation 3 trial had the highest mass pull probably due to the chemical reagent addition which activated more particles to become hydrophobic and further on ended to the froth fraction.

The behavior of few main elements in the flotation trials has been presented between Figure 9 and Figure 12. The behavior of metals are calculated based on the results from XRF, TOC and fire assay + ICP-OES (gold) analysis which was carried out on the fractions generated in the flotation experiments.

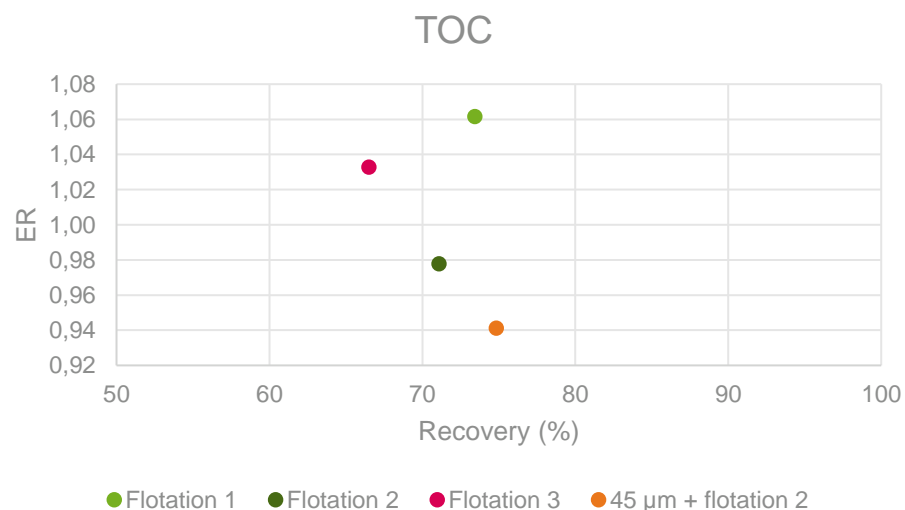


Figure 9. TOC enrichment compared to recovery in flotation trials.

The TOC did not enrich significantly to either froth or concentrate fraction (Figure 9) even though the assumption and target was to remove organics as much as possible. Clear reason for the unexpected TOC behavior has not been identified since the hypothesis is that TOC is naturally hydrophobic and should attach to air bubbles and separate to the froth fraction as it has





occurred in the flotation of crushed printed circuit board (Ogunniyi and Vermaak, 2009; Vidyadhar and Das, 2013).

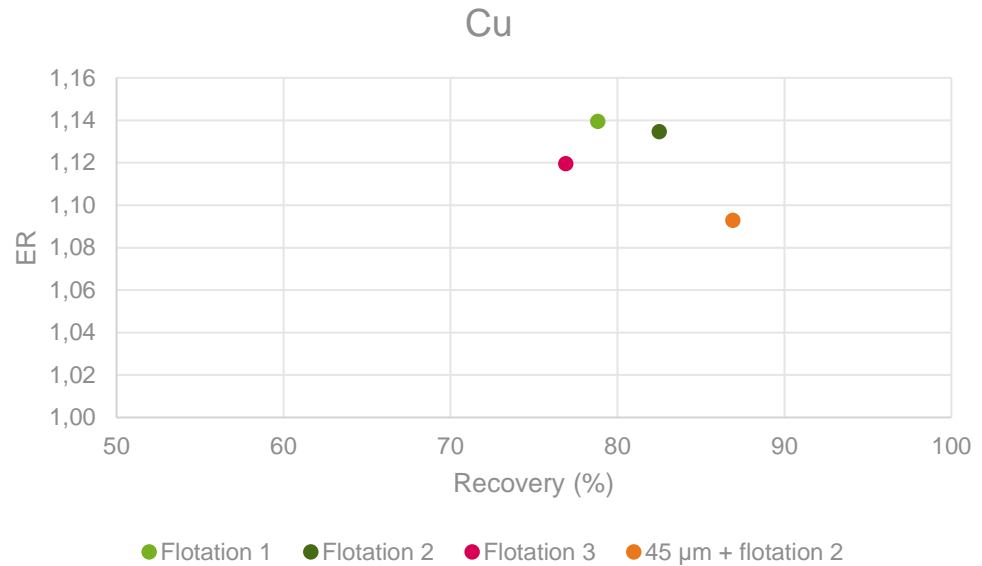


Figure 10. Copper enrichment compared to recovery in flotation trials.

Copper enriched in all flotation trials to the concentrate with rather good recoveries (Figure 10). The enrichment ratio was on a rather same level for all flotation tests. Slightly higher recovery was gained for Flotation 2 trial compared to the other two flotation trials. One reason for this could be that when the fines has been removed with sieving, the potentiality of fine copper particles migrate to the froth fraction due to the entrainment phenomenon has decreased. If even higher recovery is desired, by adding the fines fraction from the sieving to the concentrate from flotation increases the recovery up to 87 %. However, the grade of the copper naturally decreases.

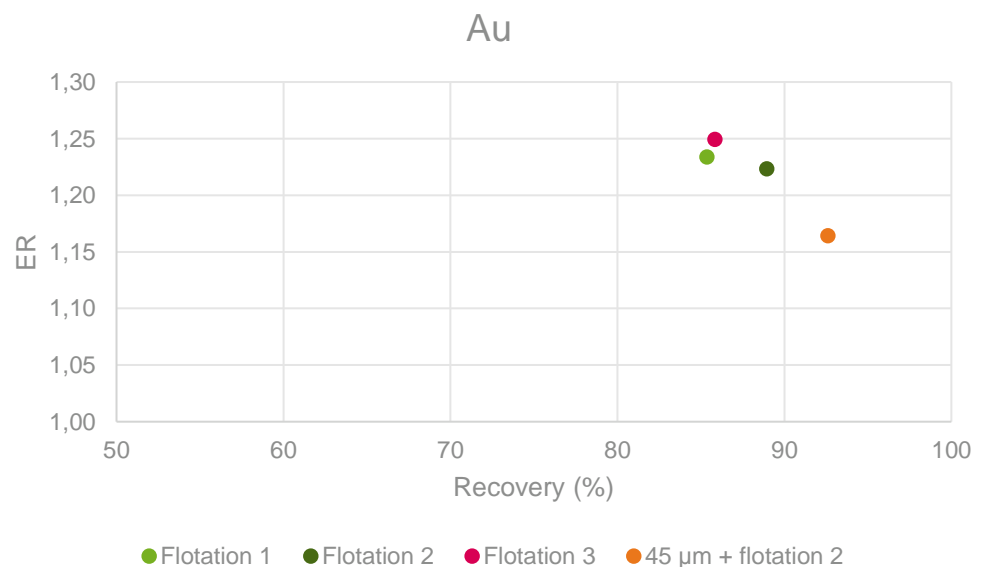


Figure 11. Gold enrichment compared to recovery in flotation trials.





The behavior of gold (Figure 11) resembled largely the behavior of copper though the separation efficiency gained higher values. Flotation 3 trial reached the highest enrichment ratio of 1.25 with around 86 % recovery. Since the value of gold is so significant, better recovery over the higher grade is preferred in order to lose the minimum amount of gold. Flotation 2 trial with sieved < 45 µm fraction generated best recovery for gold with the value of 92.6 %.

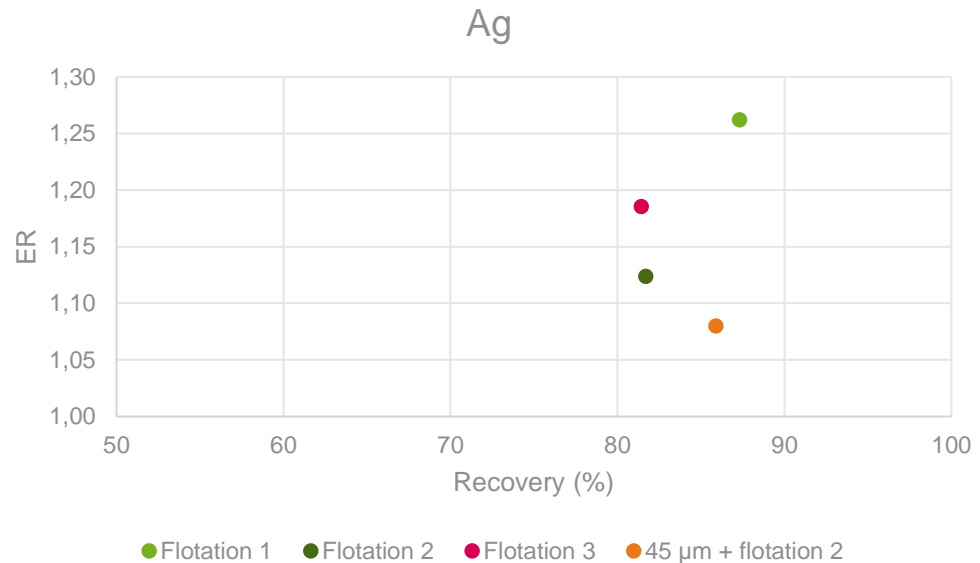


Figure 12. Silver enrichment compared to recovery in flotation trials.

Silver behavior in the flotation tests (Figure 12) varied to the other metals presented before. First, the enrichment ratios varied rather much between the trials and clear reason for this has not been identified. Secondly, sieving the fines (> 45 µm) fraction prior to flotation did not seem to improve the flotation efficiency as it did with copper and gold. Further investigations of the silver particles/agglomerates in the samples should be carried out in order to detect possible cause that could explain the behavior.

As an overall result of the flotation trials it can be summarized that metals enriched to the concentrate and sieving prior to the flotation increased the separation efficiency. Low separation efficiency for TOC was unexpected but can be to some extent be explained to the heterogeneity of the feed. The behavior of silicon and chlorine in the flotation trials were also carried out even though they are not plotted in the figure. The results from these elements show that they enrich to the froth fraction indicating inorganic materials that are not metals end mostly to the froth fraction. By carrying out flotation prior hydrometallurgy the consumption of acid and other reagents in the leaching stage can be decreased. Further, the content of some disruptive elements to leaching could possibly be decreased.





3.2.2 Hydrometallurgy

The hydrometallurgical part of the project was conducted in several leaching experiments where the metals recovery was investigated. The studied material was Dust mix. The experimental work was divided to three parts:

1. preliminary experiments with different acids, their strengths and temperatures, to understand how different elements dissolve from the dust,
2. large scale tests for validation and to obtain residual solids for following gold recovery studies,
3. gold recovery studies from native Dust mix and residual solids produced in experimental part 2.

The experimental part one consisted of 12 small scale batch leaching experiments, having the objective to evaluate the yields of iron and other base metals and to determine optimal leaching parameters, which might have positive impact on subsequent gold leaching, or reduce the environmental impact of dust. The variables were acid (nitric, hydrochloric and sulfuric acid), acid concentration (1 and 3 molar) and temperature (25°C and 75°C). The solid-liquid ratio (50 g/l), experiment duration (240 min) and the stirring speed (200 rpm) were kept constant in all of the experiments. The experiments were conducted in Erlenmeyer flask. The flask was placed on top of magnetic stirrer, which functioned also as a heater. Two samples were taken during every experiment, at 30 and 240 minutes, respectively. The leach solutions and solid leaching residuals were analyzed for elements with ICP-OES/MS. The metal content reduction was calculated based on solid residue metal content versus metal content in ingoing solid sample.

Metals content reduction for selected metals in experimental part one is presented in Figure 13 and Figure 14 for selected metals. The selection of metals is based on respective metal content in solid sample, commercial value and for the possibility to affect gold leaching. The experimental part one showed that 3 molar nitric acid was rather selective for Sn in low temperature, while 1 molar nitric acid yielded in highest Cu dissolution. Moreover, it was seen that efficient Sn removal occurs in low temperature, while efficient Cu removal requires high temperature. Three molar hydrochloric acid was seen to be effective but rather unselective leaching chemical, resulting very high removal rates for Fe, Mn, Zn, Sn, Ag and Cu with high temperature. Three molar sulfuric acid was effective in removal of Fe, Mn, Zn and Sn. For Al and Pb no selective or effective leaching parameters was found.

If the acid treatment is driven only by improving the environmental quality of the dust (i.e. to remove heavy metals), the most promising leaching method studied is 3M HCl in 75°C. By this method, only lead remains mainly in solid form. However, 3 M sulfuric acid in 75°C, having similar effects, may be more cost-effective method, due to lower price of H₂SO₄.





Selected metals content reduction in experiments in experimental part one (1/2)

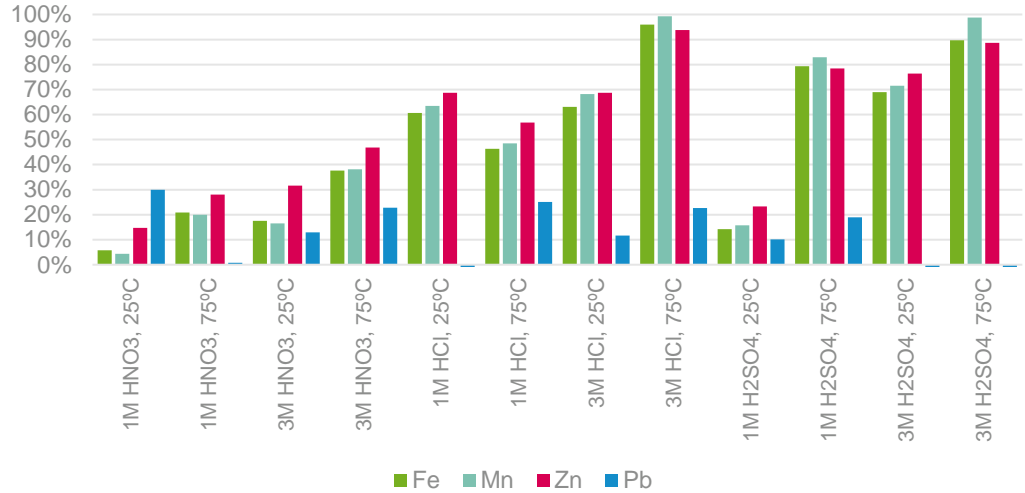


Figure 13. Selected metals content reduction in experiments in experimental part one (1/2)

Selected metals content reduction in experiments in experimental part one (2/2)

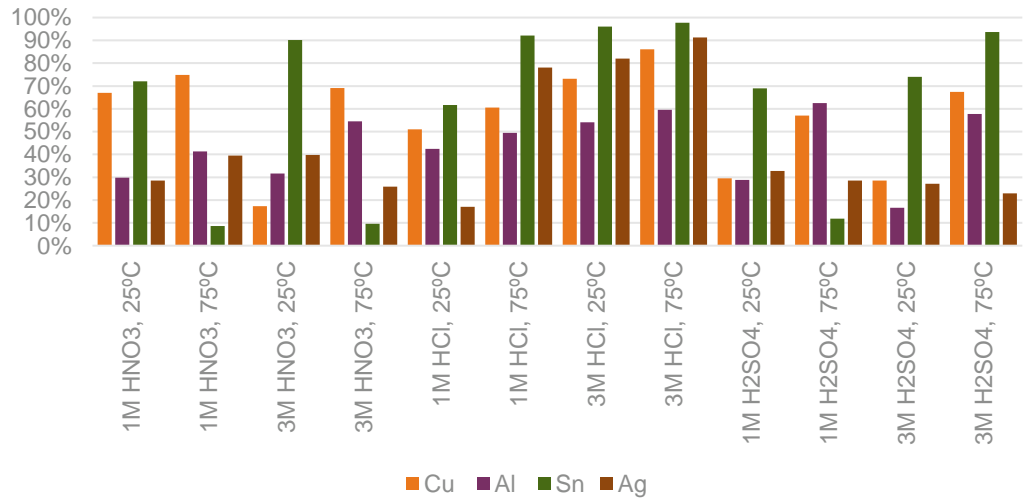


Figure 14. Selected metals content reduction in experiments in experimental part one (2/2)

The experimental part two consisted of two larger reactor batch leaching experiments and one shake flask experiment, with the objective to prepare suitable amount of solid leach residue with suitable pretreatment parameters for later gold leaching experiments. The reactor experiments were done with sulfuric acid (1 molar and 4 molar) in room temperature, with solid-liquid ratio of 200 g/l, 18 hour leaching duration and 400 rpm stirring speed. The apparatus was titanium reactor with pitch blade impeller and working volume of 5 liters. The third shake flask experiment was done with hydrochloric acid (3 molar) in 75 ° C temperature, with solid-liquid ratio of 150 g/l, 240 minutes





leaching duration and 200 rpm stirring speed. The material for all three experiments was Dust mix, and leach solution was analyzed with ICP-MS/OES at the end of the experiment. Fire Assay method was utilized for determination of gold content.

Metals content reduction for selected metals in experimental two is presented in Figure 15 for selected metals. From the results it can be stated that 1 molar sulfuric acid cannot dissolve efficiently metals from Dust mix, even with long leaching duration. By increasing sulfuric acid concentration to 4 molar, Fe, Mn, Zn and Sn are removed efficiently. Very similar results was observed with hydrochloric acid in shorter time, but with high temperature. However, hydrochloric acid dissolved efficiently also Ag and Cu. Interestingly, sulfuric acid was seen to remove Au in the experiments. The reason is hypothesized to relate on analytical or mass balance error, due to extremely heterogeneous nature of Dust mix.

Selected metals content reduction in experiments in experimental part two

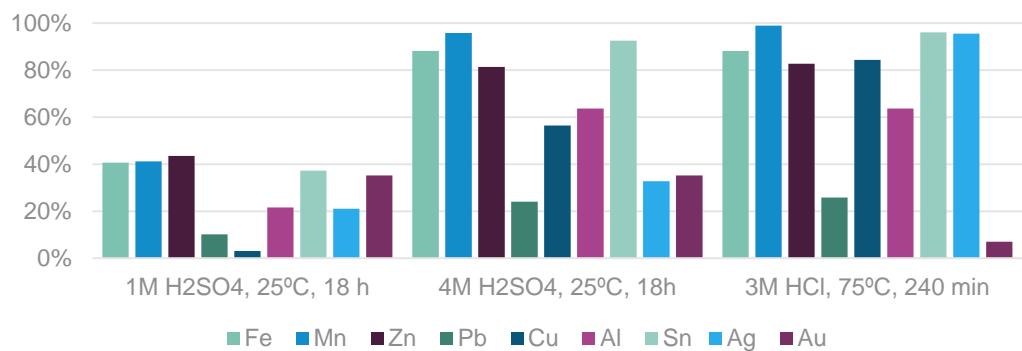


Figure 15. Selected metals content reduction in experiments in experimental part two

The experimental part three consisted of four experiments with the objective to study Au dissolution from Dust mix and solid leach residues produced in experimental part two by sulfuric and hydrochloric acid leaching. Aqua regia was used as a solvent, with 60 g/l solid-liquid ratio, 75 ° C temperature, 180 minutes leaching duration and 400 rpm stirring speed. The tests were conducted in glass reactor with PTFE coated pitch blade stirrer. Five samples were gathered during every test at times 5, 15, 30, 60 and 180 minutes. Metal yield was calculated with two methods, due to the possibility of error in solid material metal content. In the method 1, the yield was calculated based on ingoing solid sample metal content and metal content in liquid phase at certain time. In the method 2, the yield was calculated based on solid residue metal content and metal content in liquid phase at certain time.

Metal and Au content for studied solid materials acting as the feed in experimental part three is presented in Figure 16 and Table 2, respectively.





Sample content for selected metals in experiments in experimental part three

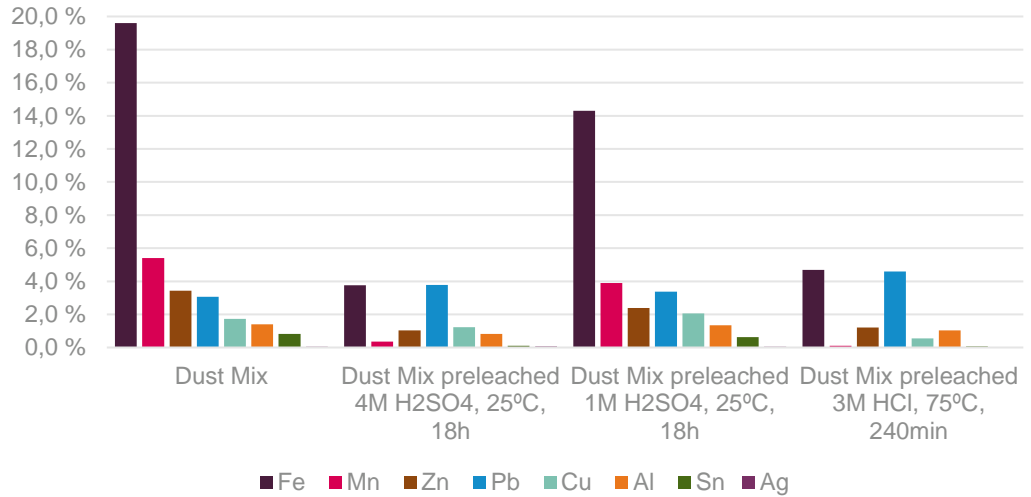


Figure 16. Elemental composition of solid feeds for experimental part 3.

Table 2. Gold content in solid samples for experimental part 3.

Experiment	Gold content in sample (ppm)
Dust Mix	13
Dust Mix preleached 4M H ₂ SO ₄ , 25°C, 18h	13
Dust Mix preleached 1M H ₂ SO ₄ , 25°C, 18h	11
Dust Mix preleached 3M HCl, 75°C, 240min	25

For the native Dust mix sample and only mildly pre-leached (1M H₂SO₄) solid sample, Aqua regia leaching caused strong dissolution for Fe, Mn, Zn, Cu, Sn and Ag (Figure 17). For the solid samples pre-treated more aggressively (4M H₂SO₄ and 3M HCl), metals dissolution was not so effective, except for Cu. For all solid samples, Au removal rate was high with Aqua regia leaching, ranging from 82 % (4M H₂SO₄ pre-treatment) to 95 % (native Dust mix sample). Au dissolution is counted here by “method 2”. From these findings, it is hypothesized that metals are not completely liberated in the solid Dust mix sample, but attached to different matrices (e.g. plastics, glass fiber). Thus, only certain amount of metals can be dissolved and final treatment with strong solvent (now Aqua regia) cannot remove all metals of the sample. This hypothesis is partly encouraged by the Au behavior, as results were very similar with both native Dust mix and pre-treated samples, respectively, showing that app. 90 % of Au is available for leaching, and app. 10 % is trapped by other matrices.





Selected metals content reduction in experiments in experimental part three

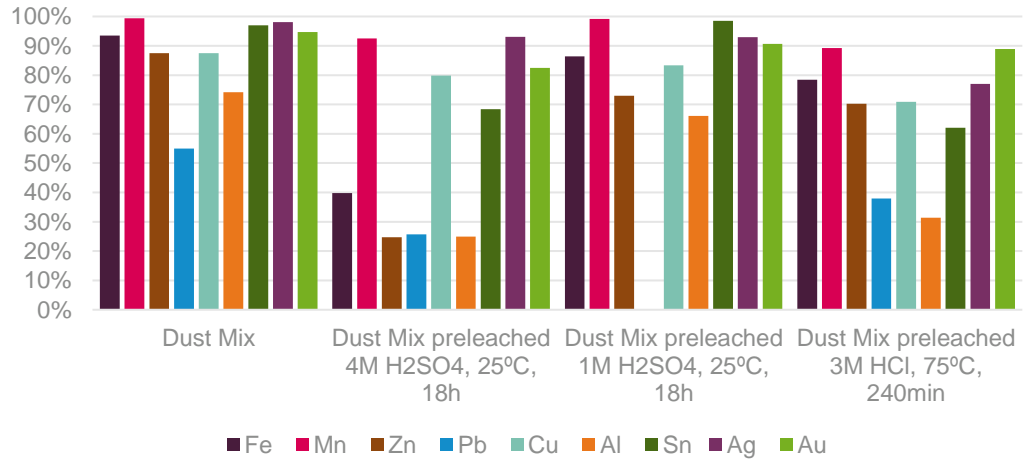


Figure 17. Selected metals content reduction in experiments in experimental part three

The leaching kinetics of elements is presented in Figure 18, Figure 19, Figure 20 and Figure 21. For native Dust mix and mildly (1M H₂SO₄) pre-leached sample, the rapid dissolution of heavy metals takes place in 15 minutes, and leaching is completed at 60 minutes. The behavior of strongly (4M H₂SO₄ and 3M HCl) pre-leached samples is different; many base metals have steady dissolution rate, which continues until the end of the experiment. The previous hypothesis of heavy metals to be trapped, may be seen here as slow dissolution of plastics, resulting in slow but steady dissolution of heavy metals.

Gold dissolution kinetics was difficult to determine, as analytic methods indicated different dissolution rates for Au, depending whether the analysis was done from solid feed and solutions (method 1) or from solid residue and solutions (method 2). This might be due to very heterogenous nature of dusts - however, gold analytics is known to be challenging. Interestingly, when determining gold dissolution by “method 1”, Au dissolution is enhanced by preliminary 4 M sulfuric acid leaching. In this case, gold was dissolving steadily throughout the experiment. With other experiments, gold dissolution was weakened in 30 minutes and finally Au concentration started to decrease.



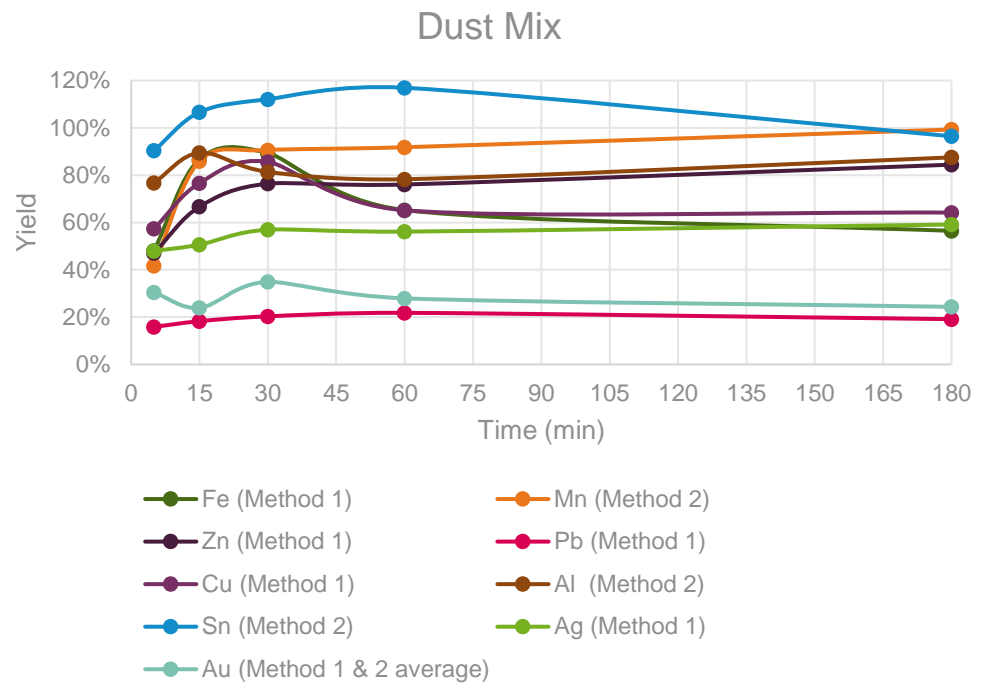


Figure 18. The yield of metals for a certain period of time in experiment "Dust Mix" in experimental part three

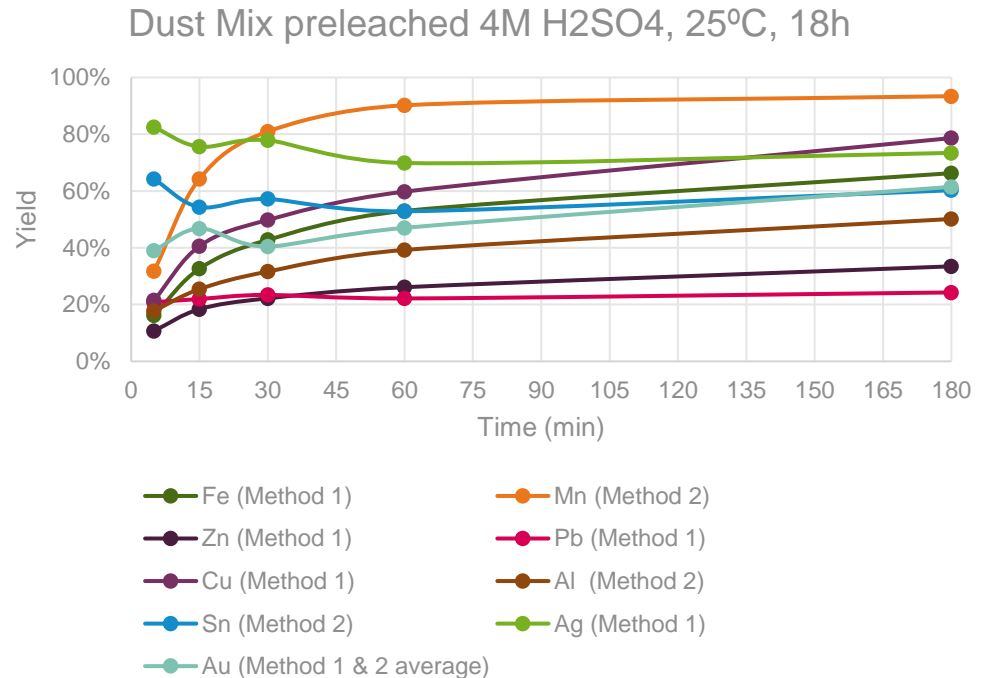


Figure 19. The yield of metals for a certain period of time in experiment "Dust Mix preleached 4M H2SO4, 25C, 18h" in experimental part three





Dust Mix preleached 1M H2SO4, 25°C, 18h

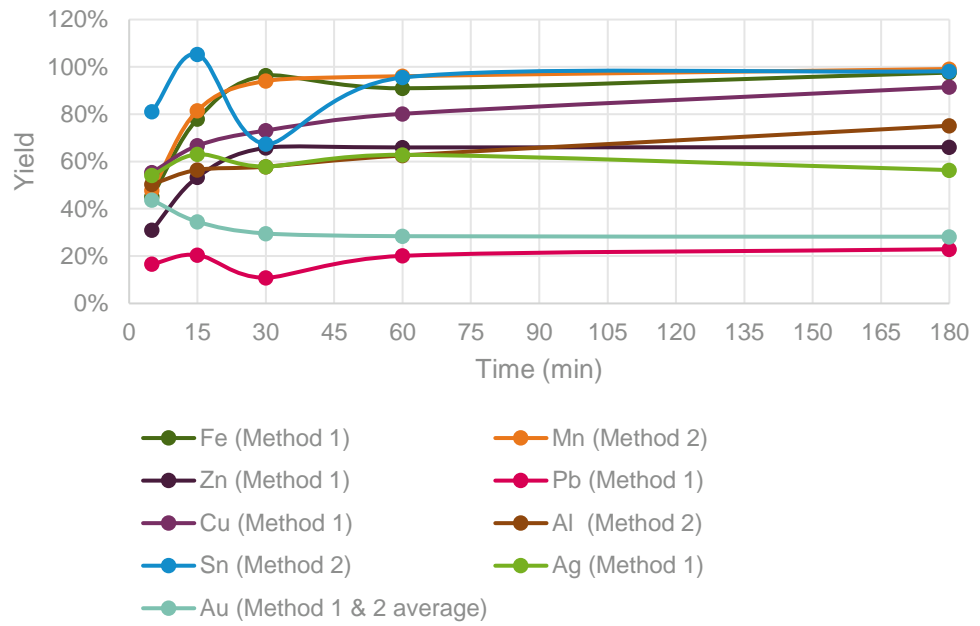


Figure 20. The yield of metals for a certain period of time in experiment "Dust Mix preleached 1M H2SO4, 25C, 18h" in experimental part three

Dust Mix preleached 3M HCl, 75°C, 240min

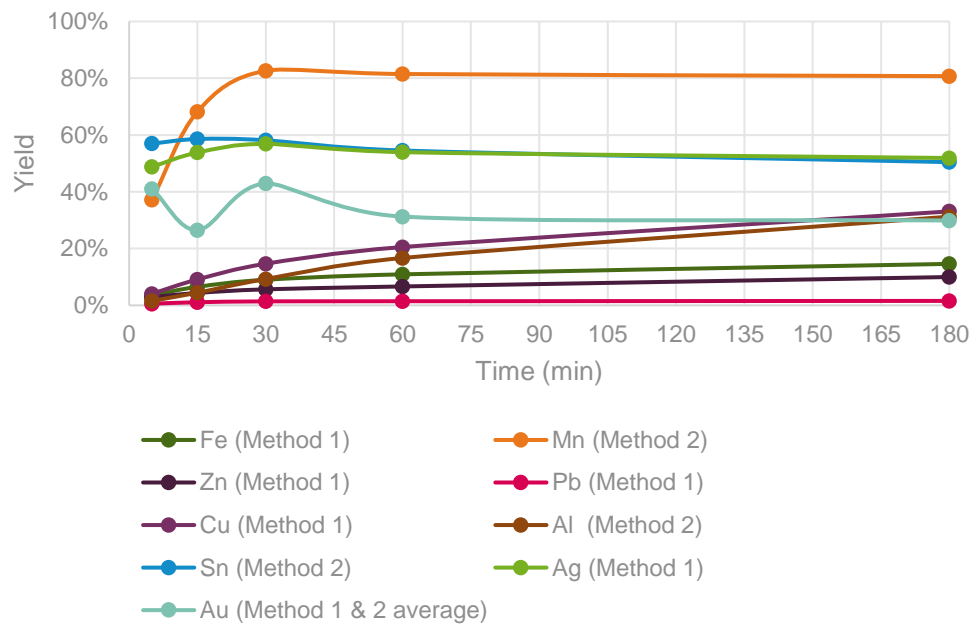


Figure 21. The yield of metals for a certain period of time in experiment "Dust Mix preleached 3M HCl, 75C, 240min" in experimental part three





4 Conclusions

The study revealed the truly heterogenic and complex nature of dusts which affects the current disposal but also on the potential further treatment options. Following main conclusion were identified in the research:

- Heterogenic and complex nature of dusts which vary in composition and particle size depending on the origin of dust in the process.
- Metals could be enriched with flotation up to 1.25 for gold
- Limited TOC removal by flotation, however in primary sieving (in preparation of "Dust Mix") roughly 60 % of TOC could be removed
- Strong HCl or H₂SO₄ leaching in elevated temperatures dissolves majority of heavy metals, except lead and some aluminum, from dusts.
- Gold can be dissolved from dusts, with 60-95% efficiency, with Aqua regia.

As a result, other treatment options than disposal could potentially be introduced for dusts from mechanical treatment. However, dust composition is directly linked to the feed of the mechanical recycling process which in this case was the low grade printed circuit board from equipment such as cathode ray tube displays and other monitors. Therefore, further research on dusts from trials with different feed materials is needed. One possible approach could also be a mixed sample composing dusts from several different trials. In addition, economical potential and profitability need to be taken into account in the future research.





5 References

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