

SUSTAINABLE WASTE-TREATMENT PROCEDURE FOR THE SPENT POTLINING (SPL) FROM ALUMINIUM PRODUCTION

TRAJNOSTNI POSTOPEK OBDELAVE ODPADKOV SPL IZ PROIZVODNJE ALUMINIJA

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Some different possible solutions to the problem of industrial waste originating from primary aluminium production (also known as SPL) have been investigated. The latest techniques to treat SPL are presented, with the highlight being flotation and chemical treatment technology. Laboratory tests confirmed that the SPL treatment model is not feasible and viable within partial solutions. Only the systematically achieved solution can be in accordance with a wide range of contemporary demands. Partly, the cause is the allotropic form of the carbon cathodes produced and partly also the impurities in the waste material that prevent an effective one-phase treatment. Laboratory investigations of reactive extraction open up the possibilities for wide SPL material utilization. The preliminary inquiry confirms that the interdisciplinary approach leads to open solutions leading to possible market interest or generated value from the waste as the main goal of a circular economy.

Keywords: spent pot line, waste treatment, flotation, alkaline-acidic treatment

Raziskane so bile nekatere možne rešitve za problem industrijskih odpadkov, ki izvirajo iz primarne proizvodnje aluminija (znani tudi kot SPL). Predstavljene so najnovejše tehnike za predelavo SPL s poudarkom na tehnologiji flotacije in kemične obdelave. Laboratorijski testi so potrdili, da model predelave SPL ne bi bil izvedljiv z delnimi rešitvami. Sistematično dosežena rešitev lahko ustreza le širokemu obsegu sodobnih zahtev. Delno je vzrok alotropna oblika proizvedenih ogljikovih katod in delno tudi nečistoče v odpadnem materialu, ki onemogočajo učinkovito enofazno obdelavo. Laboratorijske preiskave reaktivne ekstrakcije v veliki meri odpirajo možnosti za materialno izrabo SPL. Preliminarna poizvedba potrjuje, da interdisciplinaren pristop vodi k odprtim rešitvam, ki omogočajo tržni interes oziroma ustvarjeno vrednost iz odpadka, kot glavni cilj krožnega gospodarstva.

Ključne besede: izrabljene katodne obloge, obdelava odpadkov, flotacija, alkalno kislina obdelava

1 INTRODUCTION

Throughout the history of aluminium production, the industry has searched for a feasible and environmentally friendly solution for the treatment of spent potlining (SPL) as a waste product of the primary aluminium production process. SPL represents a basic part, i.e., the pot where melt of electrolyte and molten aluminium are kept during the electrolysis process, where the pot can last from 6 to 9 years. An SPL viable treatment is a global challenge for the primary aluminium industry, as is declared as a mixed hazardous waste that contains a first cut as mainly carbonaceous and a second cut as mainly refractories materials.

1.1 Origins and challenges of SPL

At the end of the electrolysis cell life, the allowed iron and silicon concentration limits in the electrolyte are exceeded and the bottom part of the cell must be

renewed. The SPL bottom part is lifted and transported to a dedicated concrete pit. In the disassembly process five major parts appear: the cryolite cover, the aluminium layer, the carbon conductor layer, the functional bricks and the metal frame. A major part of the cryolite is taken back to the process. The metal frame is renewed and new cathode blocks with chamotte and insulation bricks are lined up to form a new cathode. The spent carbon cathode and the functional bricks part remain as a waste product (**Figure 1**). The industrial naming for the carbon waste part is "first cut" as this part is the first to be stamped off. Analogically, after the first cut is collected, the bricks are on the turn as the so-called second cut. All the parts or cuts are toxic due to the impregnation with molten electrolyte during the operating period, containing leachable fluoride salts and cyanides.

The typical composition of the SPL waste is given in **Table 1**. However, it needs to be emphasized that these values can vary by up to 10 mass %.

The main challenges that the innovative solution is responding to are the following:

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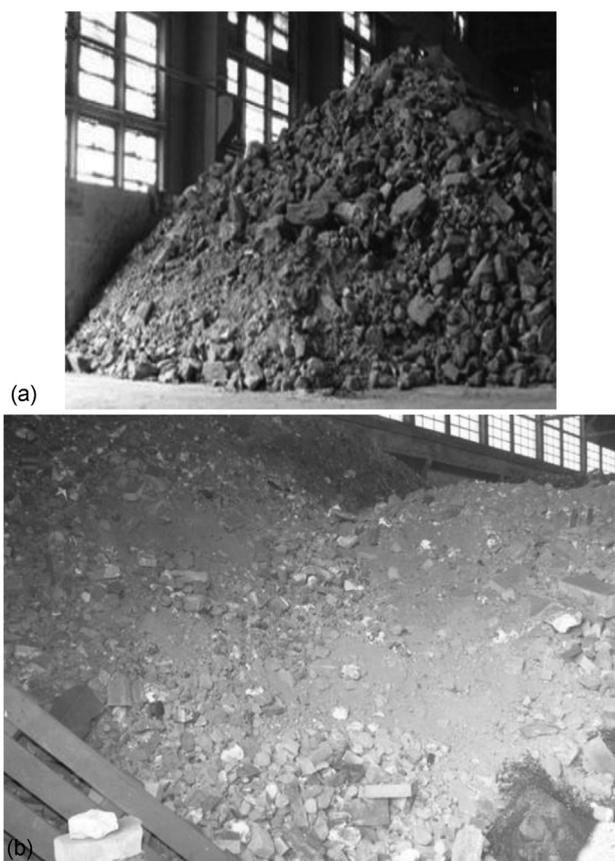


Figure 1: Typical repository of SPL waste in a warehouse: a) first-cut carbon part, b) the second cut

- A large volume of waste (problem of waste treatment, landfilling) that needs special storage and transportation requirements before landfilling and incineration
- The explosive and fire-hazardous nature of the SPL due to the potential development of explosive gases (ammonia and C₂ hydro-carbon gases) and the high burning potential (difficulty in extinguishing the fire)
- The emission of dust particles due to the high fragility of the waste material, which contains hazardous particles (cyanides, fluorides, etc.)

- The treatment of the hazardous leachates due to the high level of hazardous soluble materials (approx. 25 % (w/%) fluorides and approx. 1 % cyanides)
- The development of hazardous smoke in the case of the fire
- The corrosion properties of SPL due to the high pH (up to 13) due to the alkali metals and their oxides.
- The low social acceptance

1.2 Treatment methods summary

In principle, we can divide the SPL processing technologies into mechanical/physical, thermal and chemical. Mechanical refers to the separation of different fractions of waste, grinding, screening and other mechanical separation. For separation, specific properties of the material, such as solubility, density, chemical structure and surface properties (hydrophobicity-hydrophilicity, charge of the molecules, etc.) are used. These techniques are important primarily for the proper pre-preparation of the input material, thus achieving better efficiency in the main processes.

The best available technologies for non-ferrous metals describe many options to treat SPL: the re-use of SPL in cement production, as a carbonaceous substance in ironworks, as a secondary raw material (glass wool, salt slag) and as a substitute fuel.¹ Øye describes both the thermal and chemical methods of SPL processing, emphasizing the challenges of salt separation, which accounts for as much as 30 % of the total SPL content.² Mikša compares the use of uncleaned SPL in the cement industry and thermoelectric power plants.³

In cement plants, the carbon part is used as a substitute for fuel, while the brick part is added to the production of clinker.⁴ Consequently, there is a noticeable decrease in the process temperature in the clinker production mass, which in turn reduces the consumption of heat per kilogram of clinker produced.⁵ In ironworks, the carbon part is used as a fuel and a reducing agent. Fluorides increase the flow rate of the slag and lower the melting point, but at the same time they are also harmful (act corrosively) to the furnace wall.⁶

Table 1: Typical composition (in mass %) of SPL waste; Source: Talum d.d. Slovenia

Amorphous carbon	Graphite carbon	Mullite Al ₆ Si ₂ O ₁₃	Cryolite Na ₃ AlF ₆	Sodium fluoride NaF	Silicon dioxide SiO ₂	Cordierite (K,Mg) ₂ Al ₃ (Si ₅ AlO ₁₈)	Alumina Al ₂ O ₃	Fluorite CaF ₂	Diaoyudaote Na ₂ Al ₂ O ₃₄	Other (Ni, Al, Mg)
24,7%	19,6 %	19,0 %	12,9 %	9,6 %	4,0 %	3,8 %	2,6 %	2,0 %	1,1 %	0,6 %
44,3 %		55,7 % – Mineral part								

Table 2: Amounts of elements (in mass %) after various procedures in the carbon part of the SPL processed waste

Procedure	F (%)	Na (%)	Al (%)	Si (%)	Ca (%)	Fe (%)
Untreated SPL	7.68	5.15	5.22	5.46	0.68	0.55
Water extraction (2x)	4.85	2.41	5.48	5.88	0.81	0.62
Flotation (2xEkof)	5.78	1.87	4.44	0.71	1.24	0.25
Alkaline (FC)	2.70	2.56	2.11	0.94	2.46	0.27
Acidic (FC)	1.00	1.90	0.99	0.87	0.29	0.07

range from 5° to 90° and a 2θ step of 0.017° . Based on a qualitative analysis of the X-ray powder diffractograms with X'pert High Score Plus, the presence of several crystalline phases was identified, including phases in trace amounts. Through a quantitative analysis of the XRD diffractograms with the Rietveld method (TOPAS program), the mass proportions of the identified crystalline phases in the samples were determined. The estimated error of the calculation ranges from 3 % to 10 % for the phases with amounts below 5 %.

Scanning electron microscopy (SEM). Samples (approximately 5 mg) in the as-obtained form were positioned on aluminium carriers equipped with a carbon adhesive tape, inserted into the Zeiss SEM microscope chamber and analysed at various magnifications and acceleration voltages with three different detectors (inlens, SE2, EDS).

X-ray fluorescence (XRF) was used for the quantitative element analysis. The samples were measured on a Thermo ARL Advant'X XRF with a copper tube operated at 50 kV and 60 mA. Standardless data analysis software Thermo Scientific Uniquant (v5,49; 2008) was used for a semi-quantitative content assessment of the elements (the carbon content, determined with a TGA, was calculated for a more accurate assessment).

X-ray diffraction (XRD) was used for solid crystal phase identification using a Panalytical Cubix3 X-Ray Diffractometer equipped with an X'Celerator detector. The copper tube was operated at 50 kV and 40 mA. The conditions used were 60 min^{-1} rotation, $10\text{--}80^\circ$ 2θ , 0.011° increment, 3 s/step time per step, 3.01 min scan time, with a 0.5° fixed divergence slit. The resulting diffractograms were processed using the Panalytical HighScore+ 4.0 (2013). This software compared the measured XRD pattern with the International Centre for Diffraction Data (ICDD) database PDF-2 (2013) to find matches with known crystalline compounds.

Carbon content (C_{fix}) was measured on a Mettler Toledo TGA1 with STARe software. The sample was first heated to 950°C under inert gas for a determination of the volatiles and then burnt in the air to a constant weight. C_{fix} was calculated from the amounts of ash and volatiles.

2.2 Samples preparation

The experiments started with familiarization of the material. Many carbon-content and elemental analyses were carried in our laboratory in the past. The sample compositions were acquired with regards to the position in the cell (corner, side and bottom).

The samples were gathered from the Talum d.d. aluminium production plant. Large pieces of material were taken from the first and second cuts, in order to properly quantify the mass distribution in the cathode of the electrolytic cell (first cut 60 %, chamotte bricks 30 % and isolating bricks 10 %). The chosen bulk samples were not contaminated with cryolite crust. In the process

of crushing, some 70 % of the sample mass was obtained by sieving. The sample was crushed in a metal moon, then in a jaw crusher and finally manually in a mortar. The sample was mixed in the mixing drum in order to achieve homogeneity. The resulting material for the flotation experiment had a weight of 500 g and an average particle size of $30 \mu\text{m}$.

All the following extractions were carried out using the same methods. An L:S ratio of 10:1 was used for the extractions, the samples were stirred at a speed of 500 min^{-1} and at the end of the experimental time they were dried for 24 h in order to measure the carbon content. The sample was filtered with a blue-ribbon filter paper and dried for 24 h at 120°C to determine the residual mass.

2.3 Extraction of soluble salts

As the SPL material is impregnated with soluble salts, a leaching procedure was performed first, since the ionized salts can disturb the effect of the flotation. Technological water was chosen as a solvent to imitate the fluoride and cyanide leaching in a real industry situation. The leaching procedure was performed for a maximum time of 24 h, with the length of the individual steps after 10 min in the first hour, and then after hourly readings of the sample values.

2.4 Froth flotation

Froth flotation is a process for the selective separation of hydrophobic and hydrophilic materials. The flotation is normally performed in several stages to maximize the recovery of target minerals and the concentration of those minerals in the concentrate, while minimizing the energy input. In this work the flotation was carried out in a Denver flotation cell with two different agents.

The sample used in the froth flotation contained an average mixture of brick and carbon. It was washed twice and placed in a 1500-mL metal container of the flotation cell.

Flotation test with collector and froth reagent

For the first flotation experiment, a collector agent was chosen (sodium ethyl xanthate ($\text{C}_3\text{H}_5\text{NaOS}_2$)), used in mining as a collector for the enrichment of pyrite from quartz and galenite from sphalerite.

In 1000 mL of deionized water (L=liquid) 20 g of the sample (S=sample) was added (S:L ratio = 1:50). With a pipette, 6 droplets of collector agent were added (one droplet for approx. 3 g of sample). In the flotation cell, the slurry mixture (more properly called the *pulp*) was stirred for approx. 10 min (**Figure 3**).

Then the froth reagent (sodium lauryl sulphate, SLS) was added. When foaming, the resulting bubbles/froth were instantly pulled into a separate dish. In the froth flotation, hydrophobic particles flow to the top and sepa-



Figure 3: Denver flotation cell in operation (on the left) and sample flotation process with good adherence of carbon in the bubble-froth (on the right)

rate from the chamotte heavy particles. The process was repeated until the bubbles of the foam were transparent and lasted for approx. 1 h. Samples from the metal container (carbon part and the remaining liquid sample) were stored separately, filtered, dried for 24 h and analysed for carbon content and for elements with an XRF instrument.

Flotation test with an Ekofol reagent

To further raise the carbon content of the remaining carbon part, a second flotation test was performed. The procedure was repeated with the difference that this time more sample was processed (180 g). In addition, instead of separate by used collector and froth reagents, the Ekofol 452 GK100 collector/froth agent was used, as performed by Schönfelder.¹⁵ The process lasted 42 min and 14 droplets of the agent were used. The sample was again separated on the silicate part on the bottom and the carbon part in the froth (**Figure 3**). After double drying, the two samples were subjected to a carbon-content and an XRF elemental analysis.

2.5 Alkaline reactive extraction

Afterwards, the test with the sample obtained from the flotation tests with the Ekofol reagent followed. The water washed SPL material contained almost no soluble

salts and no silicates (bricks part); therefore, no flotation was needed anymore.

Finally, the alkali reactive leaching procedure was performed in an 1-M NaOH solution for 3 h with a L:S ratio of 10:1. Subsequently, the samples were filtered and dried to define the carbon content.

Experiments were repeated with the "first cut" samples of SPL with rather a similar compound structure to further analyse the elementary and compound composition.

2.6 Acidic reactive extraction

To reduce the remaining calcium content and further increase the carbon content an acidic reactive extraction was performed. The remaining first cut alkali-extracted sample was washed with distilled water to avoid the alkali species contamination. Then the sample was filtered, dried and stirred at a speed of 500 min⁻¹ in a 2.7-M HCl solution for 3 h in an L:S ratio of 10:1. The samples were again filtered, two-times water washed to avoid contamination, filtered and analysed.

Using a scanning electron microscope (SEM) analysis of the samples the morphology was determined.

An additional acidic test was carried out to compare the efficiency of the different types of acids. The first cut sample was subjected to the above-described water and alkali extraction. The alkali-leached sample was exposed to a 0.74-M AlCl₃ solution, in order to compare its effectiveness with respect to HCl. Finally, both samples were water washed to avoid acidic contamination and analysed.

3 RESULTS

Water extraction tests

In a 1 h (time) period, almost all the soluble salts (mostly NaF) were extracted, representing about 20 to 30 mass % of the entire sample. An additional 24 h of extraction did not increase the extracted mass significantly. In one leaching step, about 40 to 50 mass % of fluorides and cyanides were extracted. Repeated extraction removed the residual soluble components (about 4 mass % of the whole soluble salts) and decreased the pH. The XRF elemental analysis showed the greatest reduction in elements like sodium and fluoride (**Table 2**).

Flotation results

Double-dried samples after froth flotation with the collector and froth reagent were subjected to a carbon-content and an XRF elemental analysis. The carbon content of the silicate (brick) part was 4.06 mass % with the main compounds quartz (SiO₂) and mullite (Al₄Si₆O₁₀). The carbon part contains 70 mass % of carbon and other compounds, merely quartz (SiO₂) with some minor content of cryolite (Na₃AlF₆) and calcium

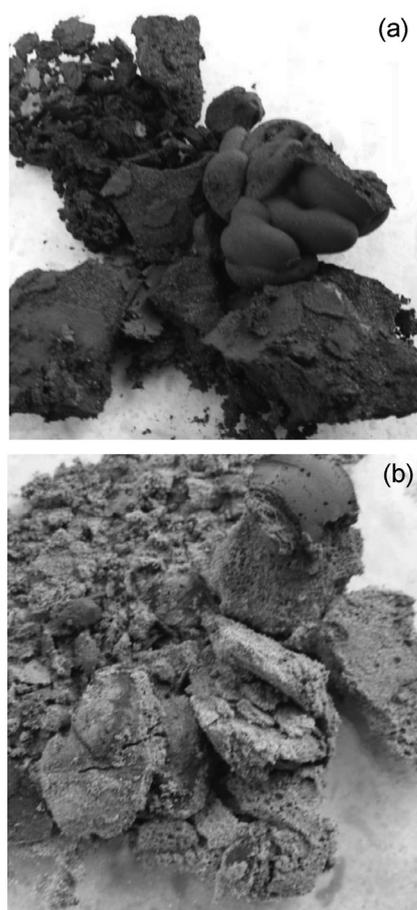


Figure 4: Obtained carbon part (left) and brick part (right) after water wash and flotation process

fluoride (CaF_2). The results are presented in **Figure 4** (picture of the separated sample) and **Figure 5** (diffractogram measurement results).

The reagent Ekofol 452 GK100, on the other hand, proved to be very effective because of the significantly better adherence of carbon (graphite and anthracite) to the surface of the bubbles. The Ekofol reagent has an

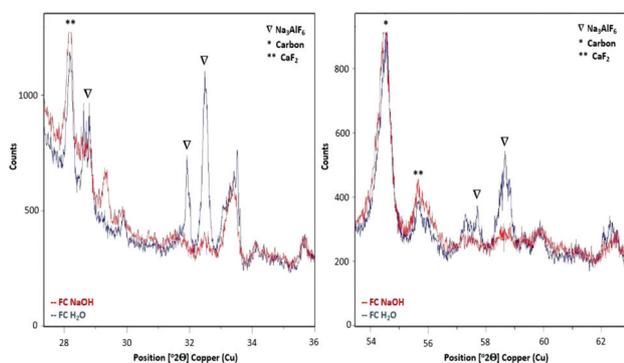


Figure 5: XRD diffractograms of an effective cryolite extraction: pixels: 29, 32, 32.5 (left picture) and 57.8, 59.6 (right picture) have a lower value on the red line (alkaline extraction step). Crystallographic data from ICDD: carbon 00-008-415, CaF_2 03-065-0535, cryolite 00-025-0772

about 4-times lower consumption and a 13-times shorter process time with respect to the collector and froth reagent.

The gained carbon part weighted 71 g and was subjected to a second-step flotation with the same procedure, using the Ekofol reagent. The process lasted 35 min and 4 Ekofol droplets were used. The second-step flotation did not noticeably increase the carbon content. The problematic compounds are the following: cryolite (Na_3AlF_6), calcium fluoride (CaF_2), gibbsite ($\text{Al}(\text{OH})_3$) and diaoyudaoite ($\text{NaAl}_{11}\text{O}_{17}$). The XRF analysis gives an elemental content of problematic species masses as follows: fluorine by 6 %, sodium by 2 % and calcium by 1 % (**Table 2**).

Alkaline extraction tests

The carbon-content analysis (C_{fix}) after the alkaline reactive extraction procedure shows a significant rise from 72 % to 82 %. The diffractogram analysis of the first-cut sample showed that with NaOH reactive extraction, removal of most of the cryolite in the waste sample was achieved (**Figure 5**). In addition, an elemental analysis made with the XRF clearly shows that elements like F, Na, and Al, that constitute cryolite, did reduce significantly. On the other hand, the calcium fraction increased (**Table 2**).

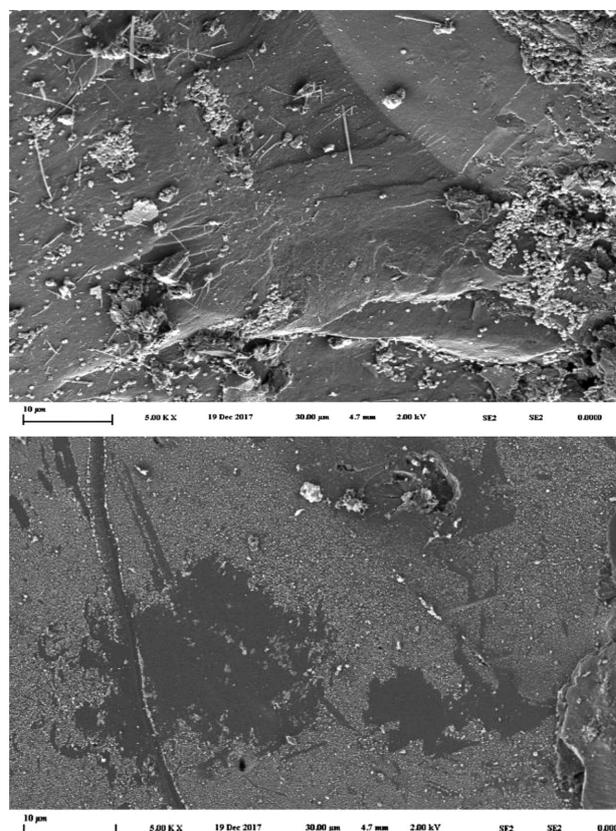


Figure 6: SEM analysis of water washed (left) and acid extracted sample (right) of the first cut

Acidic extraction tests

The results after acidic extraction showed a significant reduction of the calcium content. The sample composition obtained by qualitative and quantitative analyses with the Rietveld analysis method is as follows (w%): 3.7 % NaF, 3.5 % aluminium oxide hydrate, 1.4 % CaF₂ and 0.7 % Sodium aluminium oxide (NaAl₁₁O₁₇). The remainder was 90.7 % of graphite. The cryolite was removed completely, while the calcium fluoride and sodium aluminium oxide were significantly reduced.

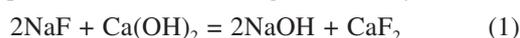
SEM results (**Figure 6**) showed that there were almost no impurities left on the surface of the cleaned sample (after the acidic leaching step). There were very little traces of spherical and needle-shaped impurities in comparison to the first-cut sample, which was water washed only.

HCl and AlCl₃ efficiency comparison

The carbon-content results in the HCl and AlCl₃ efficiency comparison test showed approximately the same values (C_{fix} around 90 mass %). In addition, the XRD diffractogram showed little changes in the species left in the sample. Both results also showed a significant drop in the CaF₂ species.

4 DISCUSSION

A four-step treatment of SPL waste has been demonstrated as a suitable approach to obtain commercial products in the form of a high content of carbon and the silicate part. Water extraction is the most effective and simplest process to dissolve the soluble NaF impurities. These are part of the filtrate, which transforms to CaF₂ and NaOH when exposed to lime (Equation 1). Both are desirable products in the metallurgical industry.



A comparison between different flotation reagents in the same conditions shows a remarkable efficiency gap between the two reagents. After the second flotation the silicon-based components like SiO₂ are significantly decreased as the insulation and firebrick parts are separated (**Table 2**). The froth-flotation efficiency is determined by a series of parameters: particle-bubble contact, particle-bubble attachment, transport between the *pulp*

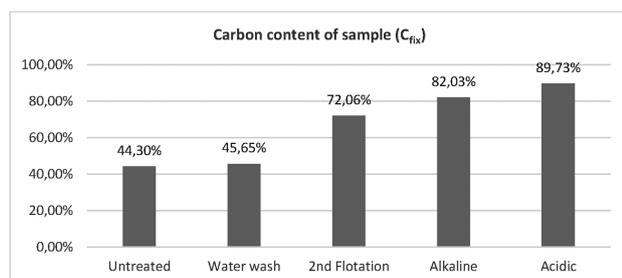


Figure 7: Fixed carbon (C_{fix}) values in different treatment solutions

and the *froth*, and froth collection into the product launder. In a conventional mechanically agitated cell (Denver flotation cell), the void fraction (i.e., the volume occupied by the air bubbles) is low (5 to 10) % and the bubble size is usually greater than 1 mm. This results in a relatively low interfacial area and a low probability of particle-bubble contact. Consequently, several cells in series are required to increase the particle residence time, thus increasing the probability of particle-bubble contact and also improving the froth flotation efficiency.

The alkali extraction was successfully performed as NaOH was effectively decomposing the sparingly soluble cryolite salt. An additional reactive extraction with acid decomposes the calcium fluoride and sodium aluminium oxide and raises the carbon content of the sample up to 90 % (**Figure 7**). The SEM images (**Figure 6**) show a clear difference in the quantity of the impurities on the surface of the treated and untreated samples.

The results showed that there is no significant difference between the acids HCl and AlCl₃ extraction as the AlCl₃ is changed to HCl. Since AlCl₃ is the more expensive compound, it is economically more appropriate to use HCl in the final extraction step.

5 CONCLUSIONS

The goal to produce purified carbon and silicate fractions from the SPL waste was achieved. The flotation results confirmed the optimal reagent choice as the separation of the silicate and carbon part was in both cases successful. The brick part is a usable product in firebrick production as it contains a reasonable part of mullite, alumina and silicates. The carbon part, on the other hand, has a high carbon content and is, therefore, a desirable product in the metallurgical industry.

Reactive extraction is proven to be an indispensable part of the process as higher purities of carbon were demanded. The by-product, i.e., the filtrate from water and alkali extraction, contains predominantly NaF, which can be transformed to usable metallurgy compounds.

A former dangerous waste is now transformed into a safe and useful product at the plant site.

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