



ARCHIVES of FOUNDRY ENGINEERING

ISSN (2299-2944)

10.24425/afe.2026.158020

Published quarterly as the organ of the Foundry Commission of the Polish Academy of Sciences

Hydrometallurgical Processing of Aluminium Drosses Using Alkaline and Acidic Leaching Media

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Received 14.06.2026; accepted in revised form 18.08.2026; available online 23.09.2026

Abstract

Aluminium dross represents an important secondary raw material containing significant amounts of metallic and oxidised aluminium. The present study investigates the hydrometallurgical processing of two aluminium drosses originating from secondary aluminium alloy production, namely rotary furnace dross and post-refining dross, representing two technologically different aluminium dross streams. The materials were characterised using X-ray fluorescence spectroscopy (XRF), X-ray diffraction (XRD), scanning electron microscopy coupled with energy-dispersive spectroscopy (SEM/EDS), and digital microscopy. Leaching experiments were performed using 5 wt.% NaOH, 5 wt.% KOH and 5 wt.% H₂SO₄ solutions at laboratory temperature. Aluminium concentrations in the leachates were determined by ICP-AES, while the solid residues were analysed by ED-XRF, XRD and SEM/EDS. The highest aluminium concentrations in the leachates were achieved using sodium hydroxide, reaching 21 800 mg·L⁻¹ for rotary furnace dross and 25 000 mg·L⁻¹ for post-refining dross. Lower aluminium concentrations (7 930–10 400 mg·L⁻¹) were obtained for potassium hydroxide and sulphuric acid. XRD analysis revealed the presence of stable phases including AlN, MgAl₂O₄, Al₂O₃, CaAl₁₂O₁₉ and CaF₂ after leaching, indicating incomplete dissolution of the original dross constituents. SEM observations confirmed substantial microstructural changes associated with partial dissolution of aluminium-bearing phases. The results indicate that, under the investigated conditions, alkaline leaching, particularly using NaOH, provides the most favourable conditions for aluminium recovery from the investigated drosses and confirms their potential as secondary raw material sources for hydrometallurgical processing.

Keywords: Aluminium dross, Secondary aluminium production, Leaching, Hydrometallurgy, Aluminium recovery

1. Introduction

Aluminium is one of the most widely used engineering materials due to its low density, high corrosion resistance and favourable mechanical properties. The increasing demand for aluminium products has led to a continuous growth in both primary and secondary aluminium production. Secondary aluminium

production is particularly important because it requires significantly lower energy consumption than primary aluminium production and contributes to the principles of circular economy and sustainable resource management [1], [2].

During aluminium melting, refining and recycling operations, significant amounts of aluminium dross are generated. Aluminium dross is a heterogeneous by-product consisting of metallic



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aluminium, aluminium oxides, nitrides, salts and various impurities originating from metallurgical processing [3]. Depending on the production route and processing conditions, the composition of aluminium dross may vary considerably. Although aluminium dross contains a substantial amount of valuable metallic and oxidic aluminium, a significant proportion of this material is still landfilled or utilised in low-value applications [4], [5], [6].

Several approaches have been developed for aluminium dross recycling, including direct remelting, pyrometallurgical processing and hydrometallurgical treatment [3], [6], [7]. Direct remelting allows recovery of metallic aluminium; however, it is often associated with oxidation losses and the formation of additional waste streams. Hydrometallurgical methods have therefore attracted increasing attention as a promising alternative for the recovery of valuable metals from aluminium-bearing wastes [3]. These methods utilise selective dissolution of aluminium compounds in alkaline or acidic media followed by subsequent recovery of dissolved species [8].

Although alkaline and acidic leaching of aluminium-bearing wastes has been widely investigated, aluminium dross cannot be regarded as a uniform secondary raw material. Its chemical and phase composition strongly depends on the melting technology, refining operations, flux additions and the stage at which the dross is generated. Consequently, results obtained for one type of dross cannot be directly transferred to another industrial dross stream. Comparative studies evaluating technologically different aluminium drosses under identical leaching conditions remain limited, particularly when both the dissolved aluminium and the composition of the resulting solid residues are considered.

The present study does not aim to introduce alkaline dissolution of aluminium as a new chemical principle. Instead, its novelty lies in the comparative evaluation of two technologically distinct industrial dross streams—rotary furnace dross and post-refining dross—using alkaline and acidic media under identical mild operating conditions. In addition to determining aluminium concentrations in the leachates, the study combines chemical, phase and microstructural characterisation of the initial materials and solid residues. This approach enables identification of the phases susceptible to dissolution as well as the chemically stable constituents that limit aluminium extraction.

NaOH and KOH were selected because both are widely used alkaline leaching reagents for aluminium-bearing secondary materials. Although both hydroxides dissolve aluminium through the formation of soluble aluminate species, recent studies have shown that they may exhibit different reaction kinetics and leaching behaviour depending on the operating conditions and the composition of the treated material. Therefore, both alkaline media were investigated in parallel under identical experimental conditions to enable their direct comparison for the investigated aluminium drosses [7] [9].

The comparison of NaOH, KOH and H₂SO₄ provides information on the influence of the leaching medium on aluminium transfer into the liquid phase and on the formation or persistence of secondary solid phases. The results are intended as a screening basis for the subsequent optimization of hydrometallurgical treatment routes adapted to the specific origin and composition of aluminium dross.

The present study investigates the hydrometallurgical behaviour of rotary furnace dross and post-refining dross generated during secondary aluminium alloy production. The aim was to compare the response of these technologically distinct dross streams to alkaline and acidic leaching and to assess their suitability as secondary raw materials for aluminium recovery. The obtained results provide information relevant to the selection and further optimisation of hydrometallurgical treatment routes for compositionally different aluminium drosses.

2. Materials and Characterisation

2.1. Investigated materials

The experimental investigation was performed using two technologically different aluminium drosses generated during secondary aluminium alloy production.

The first investigated material was rotary furnace dross generated during secondary aluminium recycling. This type of dross is formed during melting operations and typically contains metallic aluminium, aluminium oxides, nitrides and residual salt phases originating from the recycling process.

The second investigated material was post-refining dross collected after melt treatment and refining operations prior to casting. In addition to aluminium-bearing phases, this material contained residues of refining salts, modifying agents and refractory lining particles originating from metallurgical processing.

The two materials differ significantly in their origin and expected phase composition, which makes them suitable for comparative hydrometallurgical evaluation. Both materials were supplied by an industrial partner operating in the secondary aluminium alloy production sector.

The normalized elemental composition of the investigated drosses was determined by X-ray fluorescence spectroscopy (XRF) and is summarized in Table 1. The results were normalized to 100 wt.% for the detected elements, excluding carbon and oxygen.

Table 1.

Normalized elemental composition of the investigated aluminium drosses determined by XRF [wt.%]

Sample	Al	Fe	Mg	Si	Cl	Na	Ti	Cu	Zn	K	Ca	S	Others*
Rotary furnace dross	30.05	2.72	3.14	2.78	35.71	5.94	0.38	0.91	0.24	13.01	4.20	0.19	0.72
Post-refining dross	41.60	0.54	0.49	2.87	25.00	7.77	0.19	0.14	0.07	3.71	15.96	1.50	0.15

*Others represent the combined concentration of minor elements not listed individually, with a total content below 1 wt.%.

Note: The results were normalized to 100 wt.% for the elements detected by XRF. Carbon and oxygen were not included in the analysis; therefore, the reported values do not represent the absolute bulk composition of the materials.

The initial aluminium contents determined by XRF were used for calculation of the extraction efficiencies. The same sample preparation and analytical procedure were applied to both investigated drosses, providing a consistent basis for their comparative evaluation. Independent verification by complete digestion followed by ICP-OES analysis was beyond the scope of the present study but should be considered in future quantitative mass-balance investigations.

As shown in Table 1, post-refining dross contained a higher aluminium content (41.60 wt.%) than rotary furnace dross (30.05 wt.%). In contrast, rotary furnace dross exhibited higher concentrations of Fe, Mg and Cl. The elevated chlorine content in both materials is associated with residual salt phases originating from secondary aluminium processing. These differences in chemical composition are expected to influence the leaching behaviour of the investigated drosses.

The visual appearance of the investigated drosses is shown in Figs. 1 and 2. The photographs document the macroscopic appearance of the investigated industrial drosses prior to sample preparation. Although both materials exhibited a generally similar grey appearance, the rotary furnace dross consisted predominantly of smaller and more uniform agglomerates, whereas the post-refining dross showed a higher proportion of larger, more irregularly shaped agglomerates. These visual differences are consistent with their different technological origin and complement the subsequent chemical and phase characterisation.



Fig. 1. Visual appearance of the rotary furnace dross before sample preparation



Fig. 2. Visual appearance of the post-refining dross before sample preparation

2.2. Sample preparation

Prior to leaching experiments, both dross samples were dried and subsequently milled using a laboratory vibratory mill. The milling process was performed for one minute in order to reduce particle size and increase the specific surface area available for chemical reaction during leaching. Mechanical milling is commonly used as a pre-treatment step for aluminium dross processing because it reduces particle size, removes surface layers and increases the accessibility of reactive aluminium-bearing phases, thereby improving the efficiency of subsequent chemical treatment [6].

The morphology and particle size of the milled materials were evaluated using a KEYENCE VHX-6000 digital microscope in order to identify differences between the investigated drosses and to assess their suitability for subsequent hydrometallurgical processing. Representative micrographs of both drosses were obtained at a magnification of 50 $\times$ and are presented in Figs. 3 and 4.



Fig. 3. Particle morphology of rotary furnace dross (50 $\times$ magnification)



Fig. 4. Particle morphology of post-refining dross (50× magnification)

For each material, three independent particle size measurements were carried out at different locations, and 60 particles were evaluated in each measurement. The average particle size was 0.26 mm for the rotary furnace dross and 0.29 mm for the post-refining dross. The measured particle sizes ranged from 0.03 to 1.34 mm and from 0.05 to 2.32 mm, respectively. The slightly broader particle-size distribution observed for the post-refining dross may have influenced the available reaction surface during leaching.

3. Leaching experiments

3.1. Leaching Procedure

Hydrometallurgical processing of the investigated aluminium drosses was carried out using 5 wt.% aqueous NaOH, KOH and H₂SO₄ solutions. The leaching solutions were prepared using analytical-grade reagents (NaOH pellets, ≥98%; KOH pellets, ≥85%; and concentrated H₂SO₄, 96 wt.%) and deionized water. Prior to the experiments, the samples were dried, milled and homogenised as described in Section 2.

For each experiment, 125 g of aluminium dross was mixed with 1000 mL of leaching solution, corresponding to a liquid-to-solid ratio (L/S) of 8:1. The suspensions were continuously stirred throughout the experiment in order to ensure sufficient contact between the solid particles and the leaching medium.

All leaching tests were performed at laboratory temperature (approximately 20 °C) and the leaching duration was five days. After completion of leaching, the suspensions were filtered and separated into liquid and solid fractions. The solid residues were washed with distilled water and dried prior to further analyses. The liquid phase was subsequently subjected to chemical analysis, while the solid residues were used for further characterisation.

To quantify aluminium extraction, an additional series of leaching experiments was performed on a smaller scale using a reduced sample mass while maintaining the same liquid-to-solid ratio, reagent concentrations and experimental conditions. The

initial sample mass and the recovered leachate volume were recorded for each experiment, and the aluminium concentration in the leachate was determined by ICP-AES. The aluminium extraction efficiency was subsequently calculated from the mass of aluminium dissolved in the recovered leachate relative to the initial aluminium content of the corresponding dross sample determined by XRF.

Table 2 summarises the experimental conditions used during the leaching tests.

Table 2.

Experimental conditions used for leaching tests

Parameter	Value
Sample mass	125 g
Leaching solution volume	1000 ml
Leaching media	5 wt.% NaOH; 5 wt.% KOH; 5 wt.% H ₂ SO ₄
L/S ratio	8:1
Temperature	20 °C
Leaching time	5 days
Stirring	Continuous
Oxidising agent	Not used

3.2. Leaching Media

Three different leaching media were selected in order to evaluate the influence of alkaline and acidic environments on aluminium dissolution from the investigated drosses. The alkaline media consisted of 5 wt.% sodium hydroxide (NaOH) and 5 wt.% potassium hydroxide (KOH), while the acidic medium consisted of 5 wt.% sulphuric acid (H₂SO₄). A concentration of 5 wt.% was selected for all leaching media to allow direct comparison of aluminium dissolution under alkaline and acidic conditions.

These reagents were selected due to their frequent application in hydrometallurgical processing of aluminium-bearing materials. Alkaline solutions are known to promote the dissolution of metallic aluminium and amphoteric aluminium compounds, whereas acidic solutions may provide different dissolution mechanisms and reaction products.

3.3. Analytical Methods

Aluminium concentrations in the leachates were determined by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES).

The chemical composition of the solid leaching residues was evaluated by Energy Dispersive X-ray Fluorescence Spectroscopy (ED-XRF). Phase composition was determined by X-ray diffraction (XRD) using a Rigaku MiniFlex 600 diffractometer (Rigaku, Tokyo, Japan) equipped with a Cu radiation source and Ni Kβ filter. Measurements were performed in the 2θ range of 5–70° using Bragg–Brentano geometry.

Selected residues were further characterised by Scanning Electron Microscopy (SEM) coupled with Energy Dispersive

Spectroscopy (EDS) to investigate their morphology and elemental distribution after leaching.

The effectiveness of the investigated leaching media was assessed by combining the aluminium concentrations determined in the leachates with the chemical, phase and microstructural characterisation of the corresponding solid residues. This integrated analytical approach enabled evaluation of aluminium dissolution as well as identification of the stable phases remaining after leaching.

4. Results and Discussion

4.1. Aluminium Concentration in Leachates

The concentration of aluminium dissolved in the leachates is presented in Table 3.

Table 3.
Aluminium concentration in leachates

Sample	Medium	pH	Al [$\text{mg}\cdot\text{L}^{-1}$]
Rotary furnace dross	NaOH	13.02	21 800
Rotary furnace dross	KOH	13.28	8 360
Rotary furnace dross	H ₂ SO ₄	3.65	9 760
Post-refining dross	NaOH	13.11	25 000
Post-refining dross	KOH	13.25	10 400
Post-refining dross	H ₂ SO ₄	3.23	7 930

Significant differences were observed between the investigated leaching media as well as between the two types of aluminium dross. The pH values reported in Table 3 were measured in the filtrates after completion of the leaching experiments and solid-liquid separation.

The final pH values remained strongly alkaline for NaOH and KOH leaching (above 13), whereas acidic leaching with H₂SO₄ resulted in pH values between 3.2 and 3.7. Despite the similar final pH values of the NaOH and KOH leachates, the dissolved aluminium concentrations differed considerably. This indicates that the leaching efficiency was influenced primarily by the interaction between the leaching medium and the chemical and phase composition of the investigated drosses rather than by the final pH alone.

To provide a more comprehensive assessment of the leaching performance, the corresponding aluminium extraction efficiencies were calculated relative to the initial aluminium content of the investigated drosses. The calculated extraction efficiencies are presented in Table 4.

Table 4.
Aluminium extraction efficiencies obtained during leaching of the investigated aluminium drosses

Dross	Medium	Al extraction efficiency [%]
Rotary furnace dross	NaOH	36.04
Post-refining dross	NaOH	25.20
Rotary furnace dross	KOH	12.12
Post-refining dross	KOH	8.87
Rotary furnace dross	H ₂ SO ₄	19.90
Post-refining dross	H ₂ SO ₄	11.74

As shown in Table 4, NaOH achieved the highest aluminium extraction efficiency for both investigated drosses, reaching 36.0% for rotary furnace dross and 25.2% for post-refining dross. In contrast, KOH resulted in considerably lower extraction efficiencies (12.1% and 8.9%), while H₂SO₄ exhibited intermediate performance (19.9% and 11.7%).

The higher extraction efficiencies obtained with NaOH are consistent with the aluminium concentrations measured in the leachates and with the lower residual aluminium content determined in the corresponding solid residues. These findings confirm that NaOH was the most effective leaching medium under the investigated conditions.

The extraction efficiencies shown in Table 4 follow the same trend as the aluminium concentrations presented in Table 3, demonstrating good agreement between both evaluation approaches.

Differences between the two investigated drosses are attributed to their different technological origin and consequently different chemical and phase composition. Rotary furnace dross exhibited higher extraction efficiencies in all investigated leaching media, indicating that its aluminium-bearing phases were more readily dissolved under the selected experimental conditions.

For both materials, the highest aluminium concentrations were achieved using 5 wt.% NaOH solution. The aluminium concentration reached 21 800 $\text{mg}\cdot\text{L}^{-1}$ for rotary furnace dross and 25 000 $\text{mg}\cdot\text{L}^{-1}$ for post-refining dross. These results indicate that sodium hydroxide provided the most favourable conditions for aluminium dissolution among the investigated leaching media. The high efficiency of NaOH is related to the amphoteric nature of aluminium and its ability to dissolve both metallic aluminium and selected aluminium-containing phases through the formation of soluble aluminate species.

Leaching in 5 wt.% KOH resulted in lower aluminium concentrations in the leachates. The measured values reached 8 360 $\text{mg}\cdot\text{L}^{-1}$ for rotary furnace dross and 10 400 $\text{mg}\cdot\text{L}^{-1}$ for post-refining dross. Although potassium hydroxide exhibits a similar chemical behaviour to sodium hydroxide, its leaching efficiency under the selected experimental conditions was noticeably lower. The lower aluminium concentrations obtained with KOH indicate that, under the selected experimental conditions, the dissolution process was influenced not only by the alkaline character of the solution but also by the different reaction behaviour of NaOH and KOH reported in the literature, as well as by their interaction with the investigated dross composition [9]. Intermediate results were obtained using 5 wt.% H₂SO₄. The aluminium concentration in the leachate reached 9 760 $\text{mg}\cdot\text{L}^{-1}$ for rotary furnace dross and 7 930 $\text{mg}\cdot\text{L}^{-1}$ for post-refining dross. In acidic media, aluminium dissolution proceeds through a different reaction mechanism than in alkaline solutions, which may lead to the formation of secondary reaction products and consequently reduce the amount of aluminium transferred into the liquid phase. Furthermore, the acidic medium may promote the dissolution of accompanying phases containing calcium and other impurities, which can subsequently precipitate as secondary compounds instead of contributing to aluminium recovery. A comparison of both investigated materials shows that post-refining dross generally yielded higher aluminium concentrations in alkaline media, which corresponds well with its higher initial aluminium content determined by XRF analysis (41.60 wt.%). However, the calculated extraction efficiencies were consistently higher for rotary furnace dross because the dissolved aluminium represented a larger fraction of the initial aluminium

present in the material. This indicates that, under the investigated conditions, rotary furnace dross exhibited a more favourable leaching behaviour despite its lower initial aluminium content. In contrast, rotary furnace dross also exhibited slightly higher aluminium concentrations during sulphuric acid leaching. This behaviour may be associated with differences in phase composition, the presence of salt residues and the distribution of metallic and oxidic aluminium phases within the material.

Overall, the results demonstrate that the proposed evaluation approach, combining aluminium concentration with extraction efficiency, provides a more comprehensive assessment of the leaching performance. Under the investigated conditions, NaOH was identified as the most effective leaching medium for both drosses and therefore represents the most promising route for further optimisation. Although the calculated extraction efficiencies are lower than those reported for optimised alkaline leaching processes, the present study was designed as a comparative screening investigation performed under identical mild operating conditions. Therefore, the calculated efficiencies primarily serve to compare the leaching behaviour of the investigated industrial drosses rather than to maximise aluminium recovery. Future studies should focus on optimisation of the leaching conditions, including reagent concentration, temperature and leaching duration, in order to improve aluminium extraction efficiency.

The investigated leaching conditions were intentionally selected as mild laboratory conditions to enable direct comparison of two technologically different aluminium drosses. The objective of the present work was not to reproduce industrial alkaline processing routes, such as the Bayer process, or to optimise process parameters for maximum aluminium recovery. Instead, the obtained results provide a comparative basis for selecting suitable hydrometallurgical treatment routes for different industrial dross types. Future work will focus on optimisation of the most promising leaching conditions and their evaluation with respect to industrially established treatment technologies.

4.2. Chemical Composition of Leaching Residues

The chemical composition of the solid residues obtained after leaching was determined by ED-XRF analysis and is summarised in Table 5. The results reveal substantial differences in residue composition depending on the applied leaching medium.

Table 5.
Chemical composition of leaching residues [wt.%]

Sample	Medium	Al ₂ O ₃	Al	Fe	Mg
Rotary furnace dross	NaOH	59.59	31.54	1.30	5.44
Rotary furnace dross	KOH	66.40	35.14	1.10	4.07
Rotary furnace dross	H ₂ SO ₄	71.89	38.00	1.10	3.38
Post-refining dross	NaOH	63.04	33.36	0.43	0.02
Post-refining dross	KOH	65.78	34.81	0.37	0.77
Post-refining dross	H ₂ SO ₄	73.41	38.80	0.30	0.59

For both investigated materials, the lowest residual aluminium concentrations were observed after leaching in 5 wt.% NaOH solution. Rotary furnace dross exhibited an aluminium content of 31.54 wt.% after NaOH treatment, while post-refining dross contained 33.36 wt.% Al. These results correspond well with the elevated aluminium concentrations measured in the respective leachates and indicate that sodium hydroxide promoted the most effective dissolution of aluminium-bearing phases.

The residues obtained after KOH leaching showed slightly higher aluminium contents, reaching 35.14 wt.% and 34.81 wt.% for rotary furnace and post-refining dross, respectively. This observation is consistent with the lower aluminium concentrations detected in the leachates and confirms the lower leaching efficiency of potassium hydroxide under the investigated conditions.

The highest residual aluminium contents were detected after leaching in sulphuric acid solution. Aluminium concentrations reached 38.00 wt.% for rotary furnace dross and 38.80 wt.% for post-refining dross. These values suggest that a substantial proportion of aluminium-bearing phases remained in the solid residue after acidic treatment.

A similar trend was observed for Al₂O₃ equivalent values. The lowest concentrations were generally associated with NaOH leaching, whereas the highest Al₂O₃ contents were recorded after H₂SO₄ treatment. This behaviour indicates that alkaline leaching promoted the dissolution of aluminium-containing phases more effectively than the acidic medium. The residual aluminium detected after all leaching treatments indicates that a considerable proportion of aluminium remained incorporated in chemically stable phases that were not readily dissolved under the applied mild experimental conditions.

The concentrations of iron and magnesium remained relatively stable after leaching. Rotary furnace dross consistently exhibited higher Fe and Mg contents than post-refining dross, reflecting differences in the initial composition of the investigated materials. The higher magnesium content observed in rotary furnace dross may be associated with the presence of Mg-containing aluminium alloys and spinel phases formed during melting operations.

Overall, the ED-XRF results support the findings obtained from the leachate analyses and confirm that sodium hydroxide provided the most favourable conditions for aluminium removal from both investigated aluminium drosses.

Similar behaviour has been reported by authors [10], who observed efficient aluminium recovery from secondary aluminium dross using alkaline leaching and identified MgAl₂O₄ spinel as one of the phases remaining in the solid residue after treatment.

4.3. Phase composition of Leaching Residues (XRD)

The mineralogical composition of the investigated aluminium drosses and the corresponding leaching residues was evaluated by X-ray diffraction analysis. The diffractograms obtained for rotary furnace dross and post-refining dross before and after leaching are presented in Figs. 5 and 6.

The initial rotary furnace dross was characterised by the presence of metallic aluminium, halite (NaCl) and sylvite (KCl), indicating the occurrence of residual salt fluxes originating from aluminium recycling operations. After leaching, substantial

changes in phase composition were observed. The diffractograms revealed the presence of magnesium aluminate spinel (MgAl_2O_4), aluminium nitride (AlN), corundum (Al_2O_3) and gibbsite ($\text{Al}(\text{OH})_3$). The persistence of AlN , MgAl_2O_4 spinel and Al_2O_3 phases suggests that these compounds exhibit relatively high resistance towards both alkaline and acidic leaching conditions. These phases are commonly reported as major constituents of secondary aluminium drosses and their relatively high chemical stability may contribute to the limited efficiency of hydrometallurgical aluminium recovery [3] [10] [11]. The presence of gibbsite may indicate the formation of secondary aluminium hydroxide phases during the leaching process [12].

The initial post-refining dross exhibited a different mineralogical composition. Metallic aluminium and halite were identified together with calcium-containing phases originating from refining agents and refractory materials. Following leaching, the dominant phases were aluminium nitride (AlN), bayerite ($\text{Al}(\text{OH})_3$), calcium hexaaluminate ($\text{CaAl}_2\text{O}_{19}$) and fluorite (CaF_2). Compared with rotary furnace dross, the post-refining dross contained a greater proportion of calcium-bearing phases, reflecting its origin from refining and melt treatment operations. The occurrence of gibbsite in rotary furnace dross and bayerite in post-refining dross suggests differences in precipitation conditions and local chemical environments during leaching.

The most significant phase changes were observed after sulphuric acid leaching. In the case of post-refining dross, gypsum ($\text{CaSO}_4 \cdot \text{H}_2\text{O}$) was identified among the reaction products, indicating interaction between calcium-containing compounds and sulphuric acid. The formation of gypsum confirms the participation of calcium-bearing phases in the leaching reactions and demonstrates the different reaction pathway compared with alkaline leaching.

A comparison of all diffractograms shows that aluminium hydroxide phases, namely gibbsite and bayerite, were absent or only weakly represented in the initial materials but became more pronounced after leaching. This observation suggests partial dissolution of aluminium-bearing phases followed by reprecipitation in hydroxide form. At the same time, stable phases such as AlN , MgAl_2O_4 and $\text{CaAl}_2\text{O}_{19}$ remained present in the residues, indicating incomplete decomposition of the original dross constituents [11]. The persistence of these stable phases is consistent with the relatively high mass of solid residues obtained after leaching, particularly in the case of H_2SO_4 treatment. Their persistence suggests that further improvement of aluminium recovery would require either more intensive leaching conditions or additional pretreatment steps capable of disrupting these stable crystalline phases.

Overall, the XRD analysis confirmed that the applied leaching media significantly influenced the phase composition of the residues. The observed transformations correspond well with the chemical analyses of both leachates and solid residues and provide further evidence of aluminium dissolution, hydroxide formation and phase redistribution during hydrometallurgical processing.

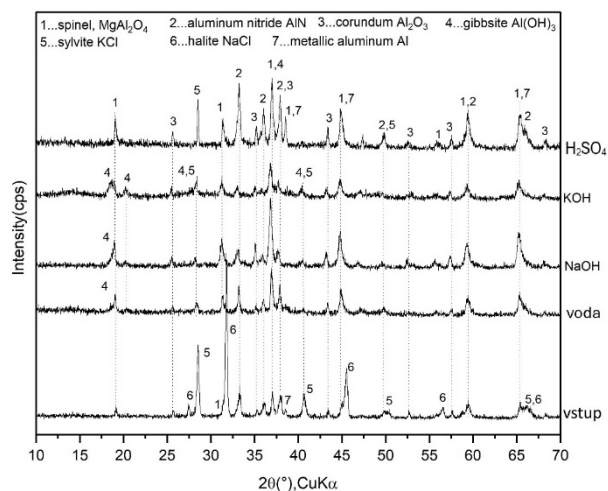


Fig. 5. Rotary furnace dross XRD diffractogram

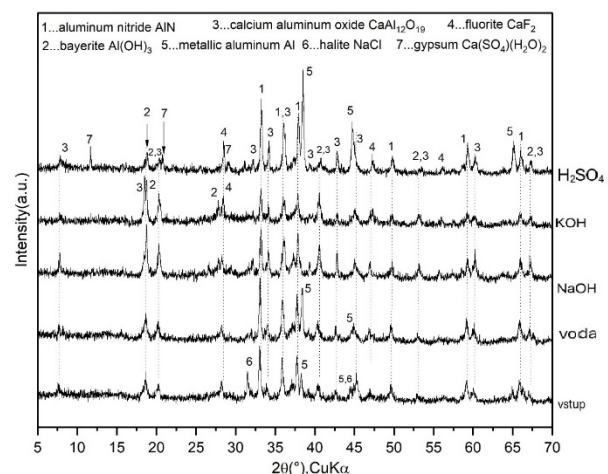


Fig. 6. Post-refining dross XRD diffractogram

4.4. Morphology of Leaching Residues

Detailed microstructural characterisation of the investigated aluminium drosses and selected leaching residues was performed using scanning electron microscopy (SEM) coupled with energy-dispersive spectroscopy (EDS). The analyses were carried out on the initial materials, as well as on residues obtained after alkaline (NaOH) and acidic (H_2SO_4) leaching. Sodium hydroxide was selected for detailed SEM/EDS investigation because it produced the highest aluminium concentrations in the leachates among all investigated leaching media.

Representative SEM micrographs and EDS spectra of the initial rotary furnace dross and post-refining dross are shown in Figs. 7 and 8. Prior to leaching, both materials exhibited a highly heterogeneous morphology consisting of particles with different sizes and shapes. EDS analysis confirmed aluminium and oxygen as the dominant elements in both materials. Minor amounts of

sodium, chlorine, magnesium, silicon and calcium were also detected, corresponding to residual salt fluxes, oxide phases and refining additives present in secondary aluminium drosses. The rotary furnace dross contained a higher proportion of magnesium-bearing phases, whereas the post-refining dross exhibited a greater contribution of calcium-containing constituents, which is consistent with the XRF and XRD analyses.

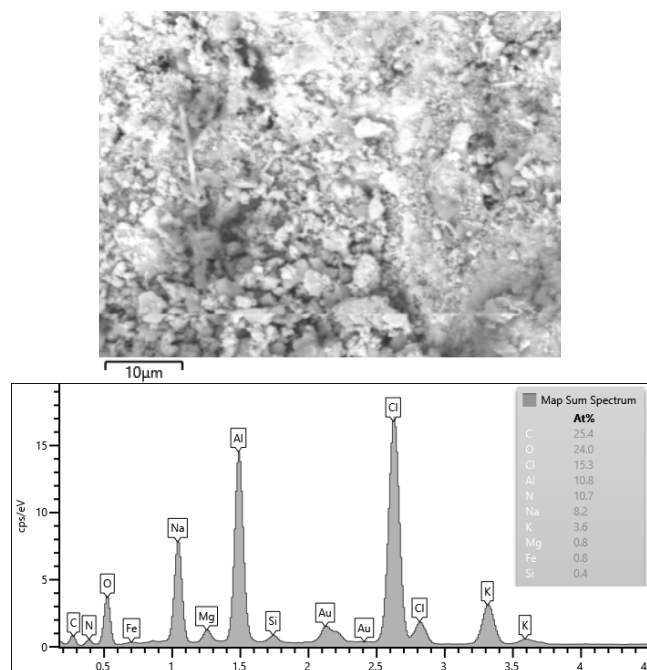


Fig. 7. Rotary furnace dross before leaching (SEM + EDS)

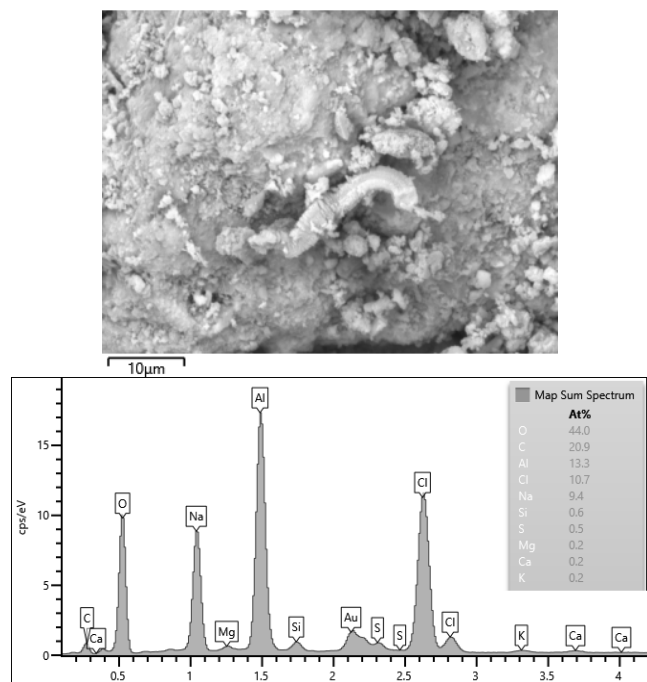


Fig. 8. Post-refining dross before leaching (SEM + EDS)

The morphology of the residues obtained after NaOH leaching is presented in Figs. 9 and 10. Compared with the initial materials, both residues exhibited noticeable surface alterations resulting from partial dissolution of aluminium-bearing phases. The residue originating from rotary furnace dross (Fig. 9) consisted of large compact particles partially covered by fine reaction products. In contrast, the residue obtained from post-refining dross (Fig. 10) displayed a more angular morphology with sharp-edged particles and fine agglomerates deposited on particle surfaces. These observations indicate that NaOH preferentially dissolved the more reactive aluminium-containing phases while leaving chemically stable compounds largely unaffected.

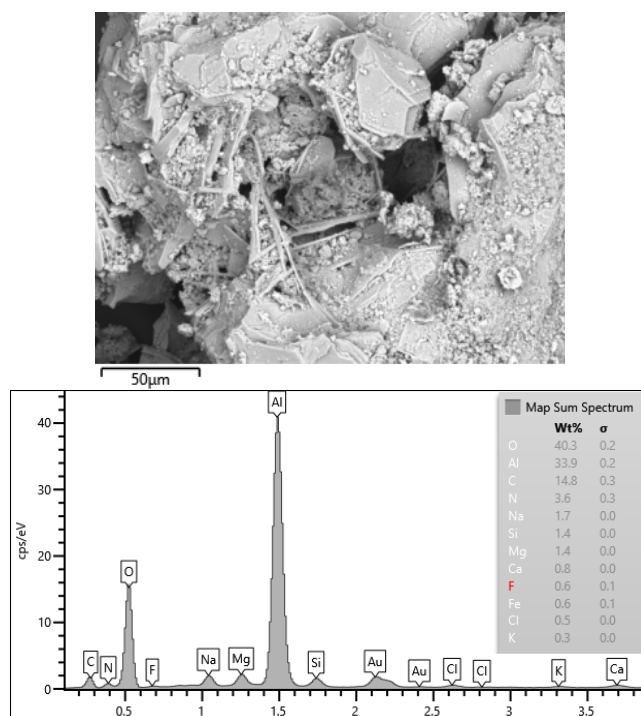


Fig. 9. Rotary furnace dross after NaOH leaching (SEM + EDS)

EDS mapping and point analyses performed on the NaOH leaching residues (Figs. 9 and 10) confirmed the presence of aluminium and oxygen as the dominant elements. Besides Al and O, smaller amounts of Mg, Ca, Na, Cl and Si were detected. The elemental distribution corresponds well with the elemental composition expected from the crystalline phases identified by XRD, particularly aluminium nitride (AlN), magnesium aluminate spinel (MgAl_2O_4), calcium hexaaluminate (CaAl_2O_9) and fluorite (CaF_2). The persistence of these phases confirms their higher resistance to alkaline dissolution under the investigated conditions. The agreement between the SEM/EDS observations and the XRD results confirms that the remaining particles were enriched in chemically stable phases after preferential dissolution of the more reactive aluminium-bearing constituents.

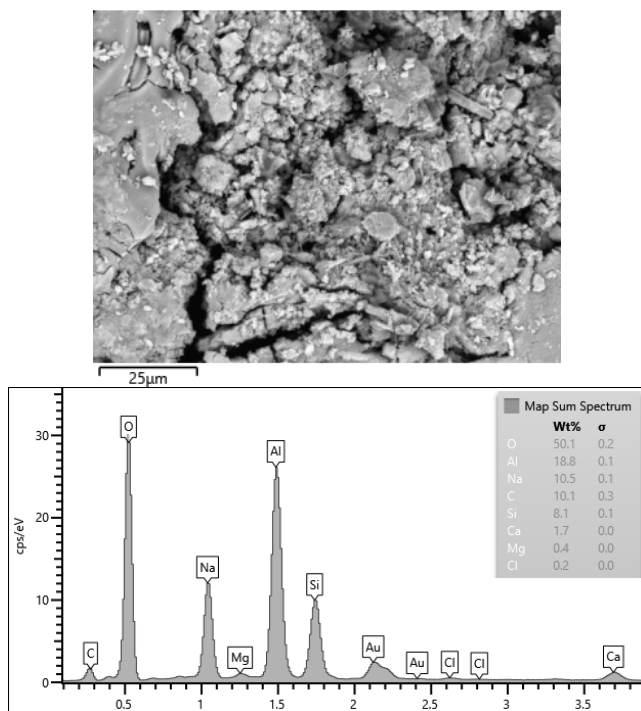


Fig. 10. Post-refining dross after NaOH leaching (SEM + EDS)

SEM micrographs of the residues obtained after sulphuric acid leaching are presented in Figs. 11 and 12. Compared with the NaOH-treated residues, the acidic leaching products exhibited a more fragmented and porous morphology. Fine agglomerated structures were observed on the particle surfaces, indicating the formation of secondary reaction products during acidic treatment. These observations are in agreement with the XRD analysis, which identified gypsum ($\text{CaSO}_4 \cdot \text{H}_2\text{O}$) in the post-refining dross residue after H_2SO_4 leaching. The formation of calcium sulphate phases demonstrates the interaction between sulphuric acid and calcium-bearing compounds originating from refining agents and refractory materials.

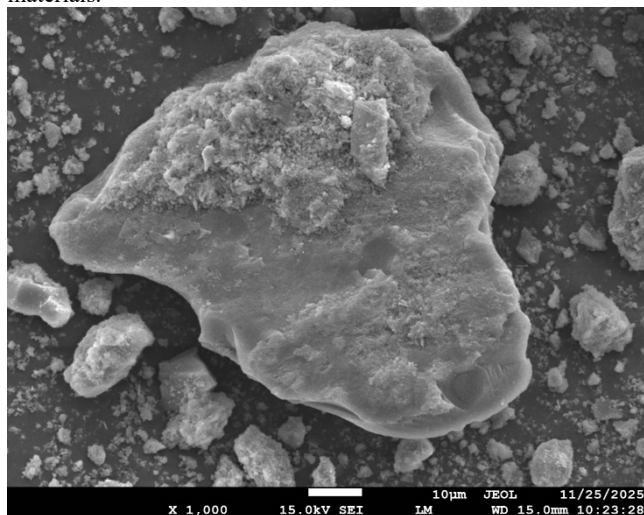


Fig. 11. Rotary furnace dross after H_2SO_4 leaching (SEM)

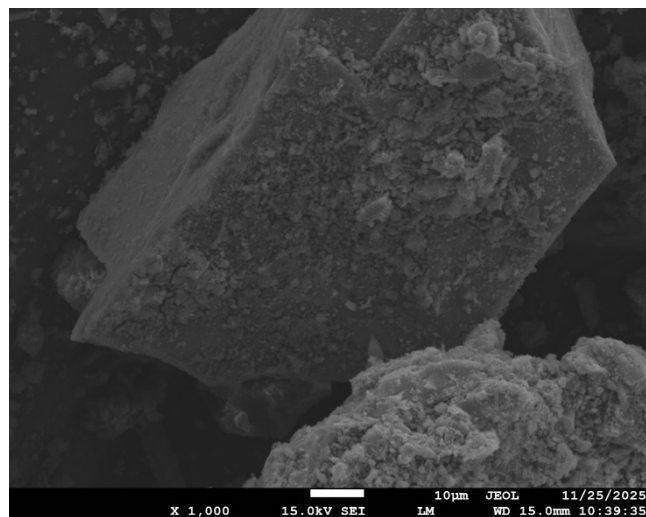


Fig. 12. Post-refining dross after H_2SO_4 leaching (SEM)

Overall, the SEM and EDS analyses support the results obtained from chemical and phase characterisation. The observed microstructural changes confirm partial dissolution of aluminium-bearing phases during leaching, while the persistence of stable phases such as AlN , MgAl_2O_4 and $\text{CaAl}_{12}\text{O}_{19}$ explains why complete aluminium extraction was not achieved under the applied experimental conditions.

5. Conclusions

Two different types of aluminium dross originating from secondary aluminium alloy processing were investigated as potential sources of secondary raw materials for hydrometallurgical recovery. Based on the obtained results, the following conclusions can be drawn:

1. The investigated rotary furnace dross and post-refining dross exhibited significantly different chemical and phase compositions, reflecting their different origins and metallurgical processing histories.
2. The highest aluminium concentrations in the leachates were achieved using 5 wt.% NaOH solution, reaching $21\,800\text{ mg}\cdot\text{L}^{-1}$ for rotary furnace dross and $25\,000\text{ mg}\cdot\text{L}^{-1}$ for post-refining dross. Lower aluminium concentrations were obtained for KOH and H_2SO_4 leaching.
3. ED-XRF analysis of the leaching residues confirmed the superior leaching performance of sodium hydroxide, which resulted in the lowest residual aluminium contents in both investigated drosses.
4. XRD analysis identified stable phases including aluminium nitride (AlN), magnesium aluminate spinel (MgAl_2O_4), corundum (Al_2O_3), calcium hexaaluminate ($\text{CaAl}_{12}\text{O}_{19}$) and fluorite (CaF_2). The persistence of these phases explains the incomplete aluminium extraction achieved under the applied experimental conditions.

5. SEM and EDS analyses revealed substantial surface alterations and partial dissolution of aluminium-bearing phases after leaching, particularly in NaOH-treated residues.
6. The obtained results demonstrate that alkaline leaching, particularly using NaOH, represents the most promising approach under the investigated conditions for aluminium recovery from the investigated drosses. The studied materials therefore represent a potentially valuable secondary raw material source for future hydrometallurgical processing.

Future research should focus on optimisation of the most promising leaching conditions identified in this study, including reagent concentration, temperature and leaching duration, as well as on subsequent recovery of dissolved aluminium from the leachates. Furthermore, future investigations should include replicate experiments, statistical evaluation, assessment of process reproducibility, and comprehensive elemental analysis of the leachates to evaluate impurity dissolution, leaching selectivity, and the purity of the recovered aluminium solutions.

Acknowledgements

This research has been supported by the project TQ15000341 (SIGMA DC2 Programme), funded with the state support of the Technology Agency of the Czech Republic (TA CR) and by projects of „Student Grant Competition” numbers SP2026/026.

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