

Physical pre-concentration and sulfidization-assisted flotation of lead-acid battery recycling slag for lead recovery

Tülin Ulucan, Nazlım İlkyaz Dinç, Şevval Yıldırım, Kerem Bölükbaş, Fırat Burat

Istanbul Technical University, Faculty of Mines, Department of Mineral Processing, 34463, Istanbul, Türkiye

Corresponding author: ulucant@itu.edu.tr (Tülin Ulucan)

Abstract: Lead-acid battery recycling slags are important secondary resources because they contain recoverable Pb-bearing phases, while their improper management poses significant environmental risks due to the toxic nature of lead. This study investigated Pb recovery through physical pre-concentration and sulfidization-assisted flotation. The slag was characterized as an Fe-rich residue containing 40.1% Fe, 4.3% Pb, and 2.0% Ca. Size-by-size chemical characterization showed that Pb and Fe were broadly distributed among the particle size fractions, indicating that classification alone was insufficient for selective Pb enrichment. Shaking table tests produced partial Pb upgrading, with heavy products containing 10.3–11.0% Pb; however, low mass yields limited Pb recovery. Magnetic separation upgraded Fe-bearing phases to 48.5% Fe in the REMS magnetic product, but Pb was also recovered in the magnetic and middling products, confirming limited Pb-Fe selectivity. Flotation performance was strongly influenced by particle size, pulp pH, collector type, and sulfidization. Although the –0.2 mm flotation feed achieved the highest Pb recovery (71.5%) with KAX, the –0.1 mm flotation feed was selected as a finer feed to evaluate reagent effects under improved liberation conditions. Natural pulp pH (approximately 10) was adopted as the operating condition. Among the tested collectors, Aerophine 3418A provided the highest Pb recovery (83.8%). Sulfidization with 1000 g/t Na₂S further increased Pb recovery to 90.2% and reduced Pb distribution in the tailing to 9.8%. Overall, sulfidization-assisted flotation proved to be an effective approach for recovering Pb from Fe-rich lead-acid battery recycling slags.

Keywords: lead-acid battery slag, Pb-Fe separation, physical pre-concentration, sulfidization-assisted flotation, sodium sulfide

1. Introduction

Lead-acid batteries (LABs) are among the oldest and most commercially mature rechargeable battery systems, yet they remain critical in modern energy storage and transportation applications. Their widespread use is primarily attributed to their low cost, reliability, high surge-current capability, tolerance to a wide range of operating conditions, and well-established manufacturing infrastructure. Although lithium-ion batteries have become dominant in portable electronics and electric vehicle applications, lead-acid batteries remain extensively used in automotive starting-lighting-ignition systems, uninterruptible power supply units, telecommunication backup systems, motive power applications, and stationary energy storage systems (May et al., 2018; U.S. Geological Survey, 2025). Consequently, lead continues to be an important metal in both primary production and secondary resource management.

Recycling spent lead-acid batteries is particularly important environmentally and economically. From an environmental perspective, lead is a toxic heavy metal, and improper disposal of lead-bearing waste can seriously contaminate soil, water, and living organisms. Economically, spent LABs are among the most important secondary lead resources because a substantial fraction of the battery mass consists of recoverable lead-bearing components. In addition to metallic grids and lead paste, recycling operations may generate intermediate residues such as slags, dusts, and fine fractions that still contain valuable Pb-bearing phases. Therefore, efficient treatment of these secondary materials is essential to

improve resource efficiency, reduce environmental impacts, and support circular economy initiatives in the lead industry (Ballantyne et al., 2018; Zhang et al., 2016).

Secondary lead production has traditionally relied on pyrometallurgical processing. Smelting-based routes are industrially established and can handle large volumes of spent battery materials; however, they also involve high-temperature operation, high energy consumption, and potential environmental emissions, particularly lead-bearing particulates and sulfur oxides. These concerns become more significant when sulfate-rich materials, such as lead paste or paste-derived residues, are directly treated without sufficient pre-treatment or desulfurization (Zhang et al., 2016; Xiong, 2022). In response, hydrometallurgical and electro-hydrometallurgical alternatives have received increasing attention because they can operate under milder conditions and may offer improved selectivity for specific lead-bearing phases (Ballantyne et al., 2018; Tan et al., 2019). Nevertheless, regardless of the downstream metallurgical route, an effective physical or physicochemical pre-treatment stage can improve process performance by reducing feed complexity and producing higher-quality concentrates or pre-concentrates.

Lead-acid battery recycling residues differ from conventional primary ores because they are highly heterogeneous secondary resources. Their composition depends on battery type, recycling technology, furnace conditions, additives, and the degree of mechanical and thermal treatment applied during processing. Lead may occur in several chemical and mineralogical forms, including metallic lead, lead oxides, lead sulfates, lead carbonates, and mixed Pb-bearing compounds. Lead paste is commonly associated with sulfate-bearing phases such as anglesite and basic lead sulfate phases, while slag-type residues may also contain Fe-rich phases, silicates, oxides, and other process-derived compounds (Xiong, 2022; Zhang et al., 2016). This complex phase assemblage directly affects the response of the material to mineral processing methods because valuable Pb-bearing particles are not always present as fully liberated grains.

Physical beneficiation methods such as classification, gravity concentration, and magnetic separation are often considered attractive pre-treatment options for recycling residues. These methods are comparatively simple, scalable, and may reduce the need for intensive chemical consumption. Classification can separate coarse and fine fractions by particle size; gravity concentration can exploit density differences between lead-bearing and gangue phases; and magnetic separation can recover or reject Fe-bearing components based on magnetic susceptibility. However, the efficiency of these methods depends strongly on the degree of liberation, particle size distribution, and the physical contrast between valuable and non-valuable phases (Leißner et al., 2013; Siddique et al., 2023). When Pb-bearing phases are finely disseminated, locked with Fe-rich particles, or present as coatings and inclusions, separation based solely on size, density, or magnetic susceptibility becomes ineffective.

The applicability of mineral-processing-based pre-concentration has increasingly been demonstrated for heterogeneous secondary resources containing valuable metals. In jewelry-processing residues, precious metals may be distributed among particles with different sizes, densities, and degrees of liberation within complex waste matrices. Gravity-based methods, including shaking-table and centrifugal concentration, have been used to upgrade Au- and Ag-bearing fractions, while flotation has been considered as a complementary recovery stage for valuable particles remaining after gravity separation (Burat et al., 2019; Ulucan et al., 2021). Similar separation limitations have also been reported for jewelry slags and sink residues, where particle size, liberation, and the association of precious-metal-bearing particles with the surrounding waste matrix strongly influenced the response to physical concentration (Ulucan et al., 2019; Ulucan et al., 2026).

Comparable physical and physicochemical separation strategies have also been applied to waste electrical and electronic equipment and battery-derived secondary resources. In fine waste printed circuit board fractions, centrifugal gravity separation followed by froth flotation was used to recover Au, Ag, and Cu, demonstrating the potential of combined beneficiation routes for finely comminuted secondary resources (Dinç et al., 2022). Particle size, shape, and geometry have likewise been shown to influence the recovery and flotation behavior of metallic particles from electronic wastes (Burat et al., 2023; Keleş et al., 2024). For spent lithium-ion batteries, integrated flowsheets involving mechanical processing, gravity concentration, froth flotation, and magnetic separation have been investigated to recover and separate metallic and non-metallic components (Mennik et al., 2023; Çuhadar et al., 2023).

Overall, these studies support integrated physical and physicochemical pre-treatment for complex secondary resources and show that separation efficiency is strongly governed by particle size, liberation, and phase association.

The Pb-Fe separation problem is particularly challenging in recycling slags because lead-bearing and iron-rich phases may coexist in composite particles. In theory, many Pb-bearing compounds have relatively high densities and could be recovered by gravity separation. In practice, however, density separation may be ineffective if Pb is not liberated or if particles contain both Pb- and Fe-bearing components. Similarly, magnetic separation can concentrate Fe-rich particles, but it may also carry Pb into magnetic products when Pb-bearing phases are physically associated with magnetic or weakly magnetic phases. Therefore, while physical methods can provide useful pre-concentration and diagnostic information, they may not always be sufficient to produce a selectively enriched Pb concentrate from complex secondary slags.

The limitations of physical methods become more pronounced in fine particle fractions. Further size reduction may improve liberation, but excessive grinding can generate slimes, increase particle entrainment, reduce gravity separation efficiency, and complicate solid-liquid separation. Fine particles also exhibit stronger surface-related effects, such as aggregation, slime coating, and changes in pulp chemistry. Under these conditions, separation performance depends on not only on physical properties but also on surface chemistry. This makes froth flotation an attractive alternative or complementary method for beneficiating fine and partially liberated Pb-bearing materials.

Froth flotation relies on differences in surface hydrophobicity and can be controlled through reagent schemes involving pH modifiers, collectors, activators, depressants, and surface-modification agents. For Pb-bearing secondary materials, flotation may provide an effective route to recover lead phases that gravity or magnetic methods cannot efficiently separate. However, direct flotation of oxidized lead minerals is generally more difficult than flotation of sulfide minerals because oxidized surfaces are often more hydrophilic and interact more weakly with conventional sulfhydryl collectors. For this reason, sulfidization is commonly applied prior to collector addition in the flotation of oxidized lead minerals. In sulfidization, reagents such as sodium sulfide introduce sulfide species into the pulp, which can react with the surface of oxidized lead minerals and form a sulfide-like surface layer, thereby enhancing collector adsorption and particle hydrophobicity (Malghan, 1986; Xie et al., 2020).

The efficiency of sulfidization-assisted flotation is highly sensitive to operating conditions. An insufficient amount of sulfide ions may fail to activate the oxidized Pb-bearing surface, whereas excessive sulfide addition may depress flotation by leaving excess sulfide species in solution or hindering effective collector adsorption. Therefore, reagent dosage, conditioning time, pulp pH, and collector type must be carefully optimized. Studies on oxidized lead minerals such as cerussite and anglesite have shown that sulfidization can significantly improve flotation performance, but only within an appropriate chemical window (Xie et al., 2020; Miao et al., 2023). This sensitivity is even more important in recycling slags, where Pb-bearing phases may coexist with Fe-rich particles, silicates, and other secondary phases that can influence reagent consumption and selectivity.

Particle size is another key parameter controlling flotation performance. Coarser particles may suffer from insufficient liberation or poor bubble attachment, whereas ultrafine particles may be lost through entrainment or reduce flotation selectivity by coating adjacent particle surfaces. Similarly, pH controls mineral surface charge, reagent speciation, collector adsorption, and the stability of sulfide species in solution. Collector type and dosage determine the degree of hydrophobicity developed on the target Pb-bearing phases, while sulfidization conditions control the formation and stability of the active surface layer required for efficient flotation. Therefore, systematically evaluating particle size, pH, collector conditions, and sulfidization is necessary to develop an effective flotation strategy for Pb recovery from lead-acid battery recycling slag.

Based on these considerations, the present study develops a flotation-based strategy to improve Pb recovery from lead-acid battery recycling slag. The flotation experiments were conducted directly on selected size fractions of the crushed original slag feed and not on concentrates obtained from the preceding gravity or magnetic separation tests. The study addresses the limitations of conventional physical beneficiation by evaluating the role of surface-chemistry-controlled flotation. Specifically, the effects of particle size, pulp pH, collector type, and sulfidization conditions on Pb recovery and Pb-Fe

separation were evaluated. The findings provide insights into the beneficiation of complex lead-bearing secondary resources and support the development of more efficient and selective recovery routes for lead-acid battery recycling slags.

2. Materials and methods

2.1. Materials

The lead-acid battery recycling slag used in this study was supplied by an industrial facility processing spent lead-acid batteries. The sample was received as a heterogeneous slag-type residue containing Pb- and Fe-bearing phases. Prior to the experimental studies, the material was homogenized and representative subsamples were prepared for particle size characterization, chemical analysis, X-ray diffraction analysis, physical pre-concentration tests, and flotation experiments.

The experimental program consisted of two independent beneficiation routes applied to representative portions of the crushed original slag feed. In the first stage, physical pre-concentration tests were performed using gravity and magnetic separation methods to evaluate the response of Pb- and Fe-bearing components to conventional physical beneficiation. In the second stage, flotation experiments were conducted to investigate the effects of particle size, pulp pH, collector type, and sulfidization on Pb recovery and Pb-Fe separation selectivity.

2.2. Sample preparation and particle size characterization

The as-received slag sample was subjected to staged comminution using jaw, cone, and roll crushers. The material was crushed to <1 mm to produce a suitable feed for subsequent characterization and beneficiation tests. After crushing, the sample was thoroughly homogenized, and representative portions were collected for dry sieve analysis and chemical characterization. The particle size distribution of the comminuted slag was determined by dry sieving. The roll-crusher product exhibited d_{80} and d_{50} values of approximately 0.56 and 0.30 mm, respectively. For the flotation experiments, representative portions of the crushed original slag were separately subjected to closed-circuit dry grinding to obtain feeds with maximum particle sizes of 0.3, 0.2, and 0.1 mm. Accordingly, the designations -0.3, -0.2, and -0.1 mm refer to cumulative undersize flotation feeds obtained by grinding the slag entirely below the respective size limits, rather than to narrow size fractions. The -1+0.3 mm and -0.3+0.053 mm fractions were used for gravity and magnetic separation, whereas the separately ground -0.3, -0.2, and -0.1 mm cumulative undersize feeds were used for flotation experiments.

2.3. Chemical analysis and mineralogical characterization

Chemical characterization was carried out on each size fraction of the roll-crusher product to determine the Pb, Fe, and Ca contents. Chemical analyses of Pb, Fe, and Ca in the feed and separation products were performed using a VARIAN/AA240FS atomic absorption spectrometer (AAS). Prior to analysis, representative samples were dried, homogenized, and pulverized. For acid digestion, approximately 0.25 g of each sample was weighed and treated with 65% nitric acid. The mixture was heated on a hot plate set at approximately 180 °C for 1 h under a watch glass, while avoiding vigorous boiling, to promote dissolution and minimize solution loss. After digestion, the solutions were cooled, diluted to the required volume, and analyzed using element-specific standard solutions after calibration. Blank solutions and element-specific calibration standards were used for quality control, and mass balance calculations were used to evaluate the consistency of the analytical results. The total feed composition and elemental distributions were calculated by mass balance from the size-by-size analytical data. These results were used to evaluate the behavior of Pb, Fe, and Ca across the particle size range and to assess whether particle size classification alone could achieve selective upgrading. In subsequent gravity, magnetic separation, and flotation tests, the products were analyzed for Pb and Fe because the study primarily focused on Pb recovery and Pb-Fe separation behavior. Sulfur analysis was carried out using an ELTRA CS580 carbon-sulfur determinator under computer-controlled operating conditions. For each measurement, approximately 50 mg of dried and homogenized sample was accurately weighed into a ceramic combustion boat and introduced into the furnace. The sample was combusted at 1350 °C,

and the sulfur content was determined directly by the instrument from the combustion gases. The measured sulfur concentrations were used to calculate the sulfur distribution in the flotation products.

The recovery and distribution values were calculated using the mass and metal content of each product. The recovery of Pb or Fe in a given product was calculated according to Eq. 1:

$$\text{Recovery (\%)} = \frac{m \cdot c}{M \cdot C} \cdot 100 \quad (1)$$

where m is the mass of the product, c is the metal content of the product, M is the mass of the feed, and C is the metal content of the feed. The same calculation approach was used for gravity separation, magnetic separation, and flotation products.

The roll-crusher product contained 40.1% Fe, 4.3% Pb, and 2.0% Ca (Table 1). The size-by-size elemental distributions show that Fe and Pb were broadly distributed across the particle-size fractions, without pronounced size-dependent enrichment. Although Ca exhibited a relatively higher content in the $-0.150+0.106$ mm fraction, its overall distribution also indicates that it was not confined to a single size class. Therefore, particle-size classification alone was not sufficient to achieve selective Pb recovery or effectively separate the Ca-bearing slag matrix from the Fe- and Pb-bearing components.

Table 1. Chemical composition and metal distribution of the roll-crusher product

Particle Size (mm)	Amount (%)	Fe		Pb		Ca	
		C (%)	D (%)	C (%)	D (%)	C (%)	D (%)
-1+0.5	30.1	45.8	34.5	5.3	36.6	1.3	19.6
-0.5+0.3	19.6	35.7	17.5	4.2	19.0	1.8	17.7
-0.3+0.212	10.1	40.9	10.3	4.4	10.3	1.4	6.8
-0.212+0.150	10.3	38.4	9.9	4.0	9.5	1.2	6.3
-0.150+0.106	7.7	35.9	6.9	3.9	6.9	6.5	25.1
-0.106+0.075	6.9	33.4	5.7	3.4	5.4	2.1	7.2
-0.075+0.053	6.4	42.0	6.7	3.7	5.4	2.4	7.5
-0.053+0.038	4.4	40.3	4.5	2.9	3.0	2.5	5.5
-0.038	4.5	37.1	4.2	3.8	4.0	1.9	4.3
Total	100.0	40.1	100.0	4.3	100.0	2.0	100.0

C: Content, D: Distribution. Only Fe, Pb, and Ca were included in the size-by-size distribution table, as these elements were consistently quantified across all fractions. Minor elements detected only in selected fractions were considered qualitatively

In addition to chemical analysis, X-ray diffraction (XRD) analysis (Fig. 1) was performed on a representative lead-acid battery recycling slag sample to identify the crystalline phases associated with Pb and Fe. The diffraction pattern was recorded using Cu K α radiation over a 2θ range of $5-70^\circ$. Phase identification was carried out by comparison with standard diffraction database patterns. The XRD results indicated metallic lead (Pb) and leadhillite $[\text{Pb}_4(\text{SO}_4)(\text{CO}_3)_2(\text{OH})_2]$ as the main Pb-bearing crystalline phases. The Fe-bearing phases were identified as potassium iron oxide ($\text{K}_2\text{Fe}_2\text{O}_5$), Mn-rich fayalite $[(\text{Fe},\text{Mn})_2\text{SiO}_4]$, and a minor Fe-rich metallic phase. The occurrence of metallic Pb together with secondary Pb sulfate-carbonate, Fe-oxide, and silicate phases confirms the mineralogical complexity of the slag. This complex phase assemblage is consistent with the limited selectivity observed during gravity and magnetic separation. In addition, identifying leadhillite indicates an oxidized Pb-bearing phase and supports sulfidization prior to flotation to improve collector adsorption and Pb recovery.

2.4. Gravity separation tests

Gravity separation tests were performed using a laboratory-scale shaking table. The tests were conducted separately on the $-1+0.3$ mm and $-0.3+0.053$ mm size fractions to evaluate the effect of particle size on density-based separation. During the experiments, the feed water and wash water flow rates were each adjusted to $3 \text{ dm}^3/\text{min}$. The stroke frequency was $300 \text{ cycles}/\text{min}$, the stroke amplitude was 15 mm , and the table inclination was 5° .

The shaking table tests produced heavy, middling, light, and fine slime products. The slime product, corresponding to the -0.053 mm material generated during the shaking table separation, was collected

separately and was not used as an independent feed fraction. Each product was dried, weighed, and chemically analyzed for Pb and Fe.

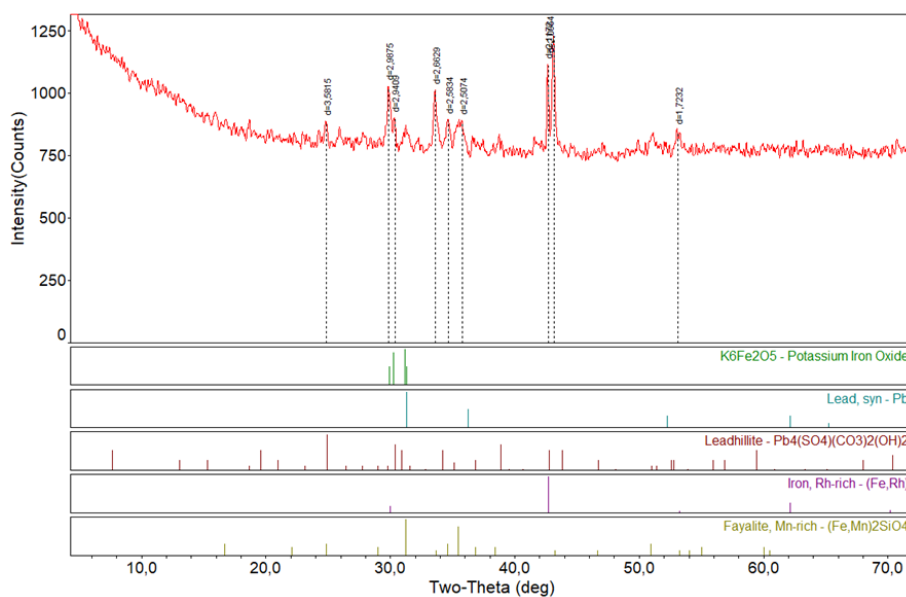


Fig. 1. X-ray diffraction pattern of the lead-acid battery recycling slag

2.5. Magnetic separation tests

Dry high-intensity magnetic separation tests were carried out using a REMS magnetic separator and a rotary disc magnetic separator. These tests were performed to evaluate the possibility of separating Fe-bearing phases from Pb-bearing components based on differences in magnetic susceptibility.

For the REMS tests (11,000 G), the $-1+0.3$ mm and $-0.3+0.053$ mm fractions were processed separately. For the $-1+0.3$ mm fraction, the magnetic and non-magnetic splitter blade angles were set to 90° and 130° , respectively, and the belt speed was 130 rpm. For the -0.3 mm fraction, the corresponding splitter blade angles were 150° and 140° , with a belt speed of 120 rpm. Magnetic, middling, and non-magnetic products were collected for each test.

Rotary disc magnetic separation tests were also performed on the same size fractions. The applied coil current was 2 A (8,500 G) for the $-1+0.3$ mm fraction and 3 A (10,500 G) for the -0.3 mm fraction. Multiple magnetic products and one non-magnetic product were obtained. All products were dried, weighed, and analyzed for Pb and Fe.

2.6. Flotation tests

Flotation experiments were conducted to investigate the effects of particle size, pulp pH, collector type, and sulfidization on recovering Pb-bearing phases from lead-acid battery recycling slag. For each flotation test, 1000 g of feed sample was used in a 2.5 dm³ Denver-type flotation cell, corresponding to a pulp density of approximately 28 wt.% solids. The impeller speed was kept constant at 1500 rpm throughout the tests. Potassium amyl xanthate (KAX), Aerophine 3418A (hereafter referred to as 3418A), and Aero 404 (A404) were tested as collectors. Methyl isobutyl carbinol (MIBC, 98% purity) was used as a frother, and sodium sulfide (Na₂S) was used as a sulfidizing reagent. Lime and sulfuric acid (95–98% purity) were used to regulate pH.

The flotation procedure consisted of staged reagent addition and concentrates collection. Collectors were added in five stages (50 + 50 + 100 + 100 + 200 g/Mg), while MIBC was added as 20 + 20 + 20 + 20 + 20 g/Mg. The stepwise addition of MIBC was applied to maintain sufficient froth stability during the five-stage flotation procedure and to compensate for frother loss associated with repeated froth removal. Each reagent addition was followed by 3 min of conditioning, after which concentrates were collected for 3 min. The total conditioning and flotation times were 15 min each. The effect of particle size was evaluated using the separately ground -0.3 , -0.2 , and -0.1 mm flotation feeds at natural pH (approximately pH 10), with KAX as the collector. Based on these results, the effect of pH was

subsequently investigated at pH 8, 10, and 11 using the -0.1 mm flotation feed. Collector performance was evaluated using KAX, 3418A, and A404 under identical reagent conditions with the -0.1 mm flotation feed at pH 10. Finally, sulfidization tests were carried out using Na₂S dosages of 500, 1000, and 1500 g/Mg with 3418A as the collector and MIBC as the frother. In the sulfidization tests, Na₂S was added before collector addition and conditioned for 10 min, then the collector was added stepwise. The concentrates and tailings obtained from the flotation tests were dried, weighed, and chemically analyzed for Pb and Fe.

Flotation performance was evaluated based on Pb grade, cumulative Pb recovery, Fe distribution to the floated products, and Pb-Fe separation selectivity. To further quantify the Pb-Fe separation performance, a simplified Pb/Fe selectivity ratio was calculated as the ratio of cumulative Pb recovery to Fe distribution to the floated products. This approach was adapted from the selectivity index concept used in flotation studies, where the selectivity between valuable and gangue components is expressed as a comparative ratio of their flotation responses (Zhou et al., 2020). In the present study, the Pb/Fe selectivity ratio was calculated using Eq. 2:

$$S(\text{Pb/Fe}) = \frac{R_{\text{Pb}}}{D_{\text{Fe}}} \quad (2)$$

where $S(\text{Pb/Fe})$ is the Pb/Fe selectivity ratio, R_{Pb} is the cumulative Pb recovery in the floated products (%), and D_{Fe} is the Fe distribution to the floated products (%). The flotation tests were performed as preliminary single-run experiments; therefore, 95% confidence intervals could not be calculated and error bars were not included in Figs. 2–5. To minimize experimental bias, all tests were carried out under controlled and identical operating conditions, and the results were interpreted as comparative trends rather than statistically optimized operating conditions.

3. Results and discussion

3.1. Particle size distribution and chemical characterization

The lead-acid battery recycling slag was crushed to below 1 mm prior to the beneficiation experiments. The particle size distribution of the roll-crusher product showed d_{80} and d_{50} values of approximately 0.56 and 0.30 mm, respectively. This distribution indicates the presence of both coarse and fine particles, which is characteristic of mechanically processed metallurgical slags containing brittle slag phases, metallic inclusions, and partially liberated particles. Accordingly, the sample was considered suitable for evaluating the influence of particle size on physical pre-concentration and flotation performance.

The size-by-size chemical characterization of the roll-crusher product (Table 1) showed an overall composition of 40.1% Fe, 4.3% Pb, and 2.0% Ca. The high Fe content confirms that the material is an Fe-rich slag, whereas Pb represents the principal recoverable metal. Calcium is mainly associated with the slag matrix formed during secondary lead smelting through interactions between fluxing agents and process impurities, leading to Fe-Ca-silicate phases (Ramus and Hawkins, 1993; Gomes et al., 2024).

The elemental distributions showed that Fe and Pb were broadly distributed among the particle size fractions without a pronounced size-dependent enrichment. Although Ca exhibited a relatively higher concentration in the -0.150+0.106 mm fraction, its distribution was likewise dispersed throughout the size range. These results indicate that particle size classification alone is insufficient to achieve selective Pb enrichment or to separate the Ca-rich slag matrix from Pb- and Fe-bearing phases. This behavior is consistent with the complex nature of lead-bearing recycling slags, where Pb may remain as residual metal or occur in oxidized, sulfate, silicate, or composite forms within the slag matrix rather than as a fully liberated phase (Salgado et al., 2024; Gomes et al., 2024).

The absence of a clear size-dependent enrichment indicates that classification alone was not sufficient for the selective concentration of Pb. XRD results showed that Pb was present as metallic lead and leadhillite, whereas Fe occurred mainly in fayalite and potassium iron oxide phases. The occurrence of these phases within the same slag matrix may explain the limited selectivity achieved by size classification. Similar behavior has been reported for Pb-bearing metallurgical slags, where crushing and classification alone provide only limited separation and are generally followed by gravity, magnetic, flotation, or hydrometallurgical processes (Baltar et al., 2012; Terzić et al., 2024). Therefore, further beneficiation steps were required to improve Pb recovery from the investigated slag.

3.2. Gravity separation performance

Shaking table tests were carried out to evaluate the applicability of density-based separation for the recovery of Pb-bearing particles from the lead-acid battery recycling slag. Since gravity separation is based on differences in particle density, it can be effective when the target metal-bearing phases are sufficiently liberated and show a clear density contrast with the gangue or slag matrix. Similar approaches have been successfully applied to Pb–Zn metallurgical slags for the preliminary upgrading of non-ferrous metal-bearing phases (Terzić et al., 2024).

As summarized in Table 2, shaking table separation resulted in moderate Pb upgrading in the heavy products for both investigated size fractions. The heavy products contained 10.3% Pb (enrichment ratio 2.2) for the $-1+0.3$ mm fraction and 11.0% Pb (enrichment ratio 2.9) for the $-0.3+0.053$ mm fraction, indicating that a portion of the Pb-bearing particles responded to density-based separation. However, the heavy product yields were only 8.1% and 4.2%, respectively, limiting the overall Pb recovery. Consequently, Pb distributions in the heavy products remained low (10.1% and 5.2%), while 45.3% and 37.6% of the Pb remained in the middling and light fractions, demonstrating that gravity separation alone was insufficient for selective Pb recovery. It should be noted that the -0.053 mm material shown in Table 2 represents the fine slime product collected during shaking table separation and not a separately tested gravity-separation feed fraction.

Table 2. Summary of shaking table separation performance for Pb recovery

Particle Size (mm)	Products	Amount (%)		Fe		Pb	
		Based on Experiment	Based on the feed	C (%)	D (%)	C (%)	D (%)
$-1+0.3$	Heavy	8.1	4.0	49.5	5.4	10.3	10.1
	Middling	44.6	22.1	37.1	22.1	4.5	24.2
	Light	47.3	23.4	34.2	21.6	3.7	21.1
	Total	100.0	49.5	36.7	49.0	4.6	55.3
$-0.3+0.053$	Heavy	4.2	1.9	49.1	2.6	11.0	5.2
	Middling	41.0	18.9	46.2	23.5	5.3	24.3
	Light	54.8	25.2	31.5	21.4	2.2	13.3
	Total	100.0	46.1	38.2	47.4	3.8	42.8
-0.053 (Slime)			4.4	30.1	3.6	1.7	1.9
Total			100.0	37.1	100.0	4.1	100.0

The limited recovery suggests incomplete liberation and strong associations between Pb-bearing particles and the Fe-rich/Ca-bearing slag matrix. Under these conditions, composite particles exhibit intermediate effective densities and therefore cannot be efficiently separated on the shaking table. Consequently, both liberated high-density Pb particles and partially liberated composite particles were present in the feed, resulting in poor separation efficiency. Similar behavior has been reported for complex Pb-bearing slags and Pb–Zn metallurgical residues, where gravity concentration provides only preliminary upgrading because valuable phases remain finely disseminated or locked within heterogeneous slag matrices (Baltar et al., 2012; Terzić et al., 2024).

Overall, the shaking table results demonstrate that gravity separation can serve as an initial pre-concentration step but is insufficient as a stand-alone process for selective Pb recovery from the investigated slag. Therefore, additional beneficiation methods based on magnetic susceptibility and surface chemistry were subsequently evaluated to improve Pb recovery and Pb–Fe separation selectivity.

3.3. Magnetic separation performance

Magnetic separation tests were performed using REMS and rotary disc magnetic separators to evaluate the feasibility of Pb–Fe separation based on differences in magnetic susceptibility. Because the slag contained approximately 40% Fe, magnetic separation was expected to concentrate Fe-bearing phases while producing a Pb-rich non-magnetic fraction. However, although Fe upgrading was achieved, Pb could not be selectively recovered in the non-magnetic products. Tables 3 and 4 present the results of the REMS and rotary disc magnetic separation tests, respectively.

As summarized in Table 3, REMS magnetic separation effectively upgraded Fe-bearing phases, producing magnetic products containing 48.5% Fe for the $-1+0.3$ mm fraction and 44.1% Fe for the $-0.3+0.053$ mm fraction. Nevertheless, Pb was predominantly recovered in the magnetic and middling products rather than in the non-magnetic fraction. For the $-1+0.3$ mm fraction, 55.1% of Pb was distributed to the combined magnetic and middling products, whereas only 0.9% reported to the non-magnetic product. Similar behavior was observed for the $-0.3+0.053$ mm fraction, with 43.6% and 0.5% Pb reporting to the magnetic/middling and non-magnetic products, respectively. The non-magnetic product yields (1.1% and 0.5%) were also very low, making their contribution to overall Pb recovery negligible. These results indicate limited Pb-Fe selectivity and suggest that Pb-bearing phases were likely associated with magnetic Fe-bearing particles owing to incomplete liberation.

Table 3. REMS magnetic separation results of the lead-acid battery recycling slag

Particle Size (mm)	Amount (%)	Products	Amount (%)	Based on the feed (%)	Fe		Pb	
					C (%)	D (%)	C (%)	D (%)
$-1+0.3$	54.6	Magnetic	39.0	21.3	48.5	25.3	6.9	32.8
		Middling	59.9	32.7	36.2	28.9	3.1	22.3
		Non-magnetic	1.1	0.6	24.4	0.4	6.6	0.9
		Total	100.0	54.6	40.9	54.6	4.6	55.9
$-0.3+0.053$	45.4	Magnetic	43.7	19.8	44.1	21.4	4.7	20.6
		Middling	55.8	25.3	38.6	23.9	4.1	23.0
		Non-magnetic	0.5	0.2	11.5	0.1	9.7	0.5
		Total	100.0	45.4	40.9	45.4	4.4	44.1
Total Feed	100.0			100.0	40.9	100.0	4.5	100.0

The rotary disc magnetic separation tests (Table 4) confirmed the limited Pb-Fe selectivity observed in REMS separation. In the $-1+0.3$ mm fraction, most of the Pb reported to the first magnetic product, with a Pb distribution of 84.9% and a Pb grade of 6.4%. However, this product also contained a substantial proportion of Fe, corresponding to 62.1% of the Fe distribution. For the $-0.3+0.053$ mm fraction, Pb was mainly distributed between the first and second magnetic products, with Pb distributions of 59.3% and 38.3%, respectively. Although the non-magnetic product of the $-0.3+0.053$ mm fraction showed a high Pb grade of 15.7%, its mass yield was only 0.5%, resulting in a Pb distribution of only 2.0%. Therefore, the non-magnetic products could not be considered meaningful Pb concentrates, despite their locally high Pb grades.

Table 4. Rotary disc magnetic separation results of the lead-acid battery recycling slag

Particle Size (mm)	Products	Amount (%)	Fe		Pb	
			C (%)	D (%)	C (%)	D (%)
$-1+0.3$	Magnetic 1	53.5	43.7	62.1	6.4	84.9
	Magnetic 2	4.8	26.3	3.4	3.4	4.0
	Magnetic 3	30.5	35.0	28.3	0.9	6.6
	Magnetic 4	10.2	22.6	6.1	1.1	2.7
	Non-magnetic	1.1	5.2	0.1	6.7	1.8
	Total	100.0	37.6	100.0	4.0	100.0
$-0.3+0.053$	Magnetic 1	41.4	44.0	46.9	5.8	59.3
	Magnetic 2	57.6	35.5	52.7	2.7	38.3
	Magnetic 3	0.5	27.7	0.4	3.7	0.5
	Non-magnetic	0.5	4.8	0.1	15.7	2.0
	Total	100.0	38.8	100.0	4.1	100.0

The reporting of Pb and Fe suggests that Pb-bearing phases were likely associated with Fe-rich particles, which limited the selectivity of magnetic separation. Similar behavior has been reported for complex Pb-bearing metallurgical slags, where mineral liberation and phase associations control separation efficiency (Terzić et al., 2024; Baltar et al., 2012).

Overall, both magnetic separation techniques successfully upgraded Fe-bearing phases but failed to recover Pb selectively. The absence of distinct size-dependent enrichment, together with the poor response to gravity and magnetic separation, suggests that Pb-bearing phases are likely associated with Fe-rich and Ca-bearing matrices. Consequently, magnetic separation alone is not an effective stand-alone process for this slag. These findings highlight the need for subsequent surface-chemistry-based separation, particularly sulfidization-assisted flotation, to improve Pb recovery and Pb-Fe selectivity.

3.4. Flotation performance

The results from classification, gravity concentration, and magnetic separation consistently indicate that Pb-Fe separation was primarily limited by incomplete liberation and the formation of composite particles. Although these physical pre-concentration methods partially upgraded the feed, none produced a selectively enriched Pb product. Accordingly, flotation was investigated as a complementary beneficiation method because it exploits differences in surface properties rather than density or magnetic susceptibility. The effects of particle size, pulp pH, collector type, and sulfidization were evaluated sequentially to determine the optimum conditions for Pb recovery and Pb-Fe separation. Since the flotation tests were conducted as preliminary single-run experiments, the flotation results are discussed as comparative trends under controlled experimental conditions.

The effect of particle size on Pb flotation was investigated at natural pH (approximately pH 10) using the separately ground -0.1, -0.2, and -0.3 mm flotation feeds. KAX and MIBC were used as the collector and frother, respectively. Flotation products were collected in five successive stages, and the cumulative flotation performance is presented in Fig. 2 in terms of floated mass, Pb distribution, and Fe distribution.

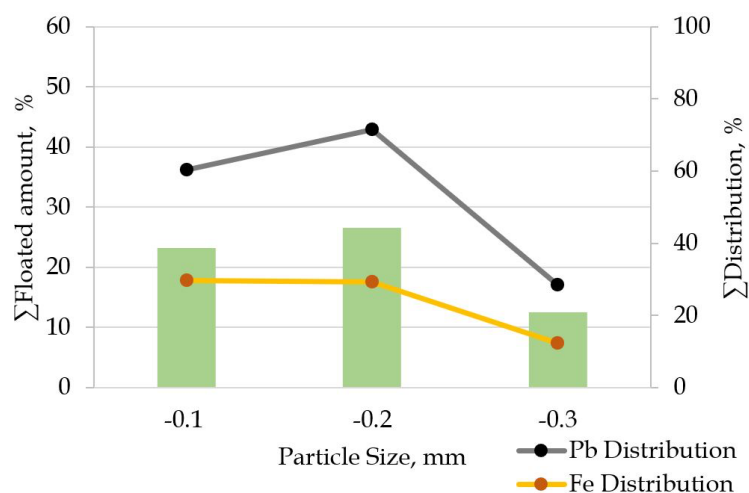


Fig. 2. Effect of particle size on Pb flotation performance at natural pH

The results show that particle size significantly affected Pb flotation performance (Fig. 2). The -0.2 mm flotation feed produced the highest Pb recovery, with 71.5% Pb recovered in 26.6% floated mass, whereas the -0.1 mm flotation feed recovered 60.3% Pb at a floated mass of 23.3%. In contrast, the -0.3 mm flotation feed showed the poorest flotation performance, with only 28.5% Pb recovery and 71.5% of Pb remaining in the tailing. These results indicate that an intermediate particle size provided the most favorable balance between mineral liberation and flotation efficiency. The calculated Pb/Fe selectivity ratios for the -0.1, -0.2, and -0.3 mm flotation feeds were 2.03, 2.44, and 2.30, respectively. These values were calculated as follows: -0.1 mm = $60.3/29.7 = 2.03$, -0.2 mm = $71.5/29.3 = 2.44$, and -0.3 mm = $28.5/12.4 = 2.30$. The -0.2 mm flotation feed therefore provided both the highest cumulative Pb recovery and the highest Pb/Fe selectivity ratio in the preliminary particle size tests. However, the -0.1 mm flotation feed was selected for the subsequent reagent optimization tests as a finer feed to evaluate reagent effects under improved liberation conditions.

Although the -0.2 mm flotation feed achieved the highest Pb recovery, the -0.1 mm flotation feed was selected for the subsequent flotation experiments to investigate reagent effects under conditions of greater particle liberation. This approach minimized the influence of incomplete liberation and enabled more reliable evaluation of the effects of pH, collector type, and sulfidization. Nevertheless, the considerable Pb remaining in the tailings and the simultaneous recovery of Fe-bearing particles indicate that particle size reduction alone was insufficient to achieve effective Pb-Fe separation.

This particle-size effect is consistent with the fundamental principles of flotation, where particle size controls liberation, bubble-particle collision, attachment, detachment, and entrainment (Ralston et al., 1999; Wang et al., 2015). Coarser or insufficiently liberated composite particles may be difficult to recover because of poor liberation and a higher probability of detachment, whereas very fine particles may show reduced collision efficiency and increased entrainment. Therefore, selecting the -0.1 mm flotation feed for subsequent tests should be considered a liberation-oriented experimental choice rather than the condition that produced the highest Pb recovery in the preliminary size tests. Miettinen et al. (2010) and Wills and Finch (2015) have discussed similar particle-size limitations in flotation.

The effect of pulp pH on Pb flotation was evaluated using the -0.1 mm flotation feed with KAX as the collector and MIBC as the frother. Fig. 3 summarizes the cumulative flotation performance obtained at pH 8, natural pH (approximately pH 10), and pH 11. As shown in Fig. 3, pH strongly influenced flotation performance. The highest Pb recovery (84.8%) was obtained at pH 8, where 36.0% of the feed was reported to the floated products. However, this condition also resulted in the highest Fe recovery (42.7%), indicating limited Pb-Fe selectivity. At natural pH (approximately pH 10), Pb recovery decreased to 60.3%, while Fe distribution to the floated products decreased to 29.7%, resulting in a lower mass pull and improved Pb-Fe selectivity. Increasing the pH to 11 further decreased Fe distribution to the floated products (22.5%), but Pb recovery remained essentially unchanged (59.3%), indicating that additional alkalinity provided no significant benefit. The calculated Pb/Fe selectivity ratios were 1.99, 2.03, and 2.64 at pH 8, 10, and 11, respectively. Although pH 11 provided the highest Pb/Fe selectivity ratio, natural pH was selected as a practical baseline condition because it avoided pH adjustment while maintaining a reasonable balance among Pb recovery, mass pull, Fe distribution, and reagent consumption.

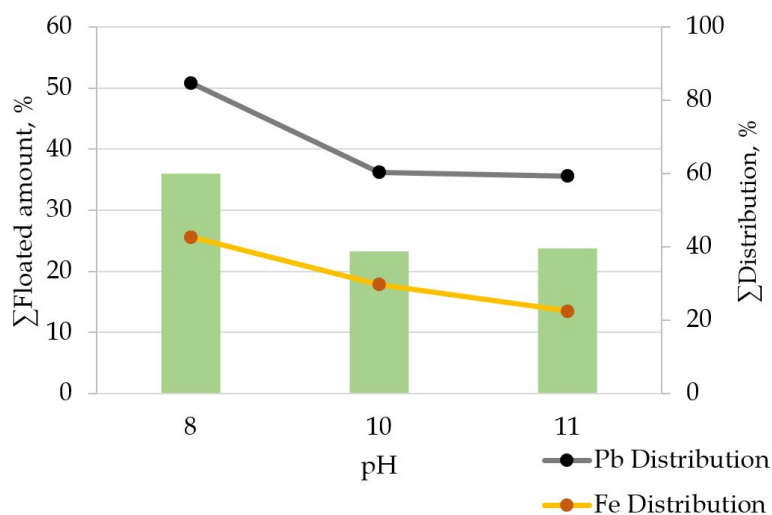


Fig. 3. Effect of pH on Pb flotation performance of the -0.1 mm flotation feed using KAX as collector

Although pH 8 produced the highest Pb recovery, natural pH was selected for the subsequent flotation experiments because it eliminated the need for pH adjustment while maintaining a reasonable balance among Pb recovery, Fe recovery, and reagent consumption. Consequently, pH 10 was considered the most practical operating condition for collector and sulfidization optimization. This observation highlights that maximizing Pb recovery alone is insufficient for process optimization; operational simplicity, reagent consumption, and Pb-Fe selectivity should also be considered when selecting flotation conditions.

The observed pH dependence may be attributed to changes in mineral surface charge, hydrolysis reactions on Pb- and Fe-bearing surfaces, and the adsorption behavior of KAX. The higher Pb and Fe recoveries obtained at pH 8 indicate a less selective flotation response, whereas increasing the pH reduced Fe recovery and limited the simultaneous flotation of Fe-bearing particles.

The effect of collector type on Pb flotation was investigated using the -0.1 mm flotation feed at natural pH (approximately pH 10). KAX, A404, and 3418A were evaluated under identical flotation conditions using a total collector dosage of 500 g/Mg, while MIBC (100 g/Mg) was used as the frother. Fig. 4 presents the cumulative flotation results.

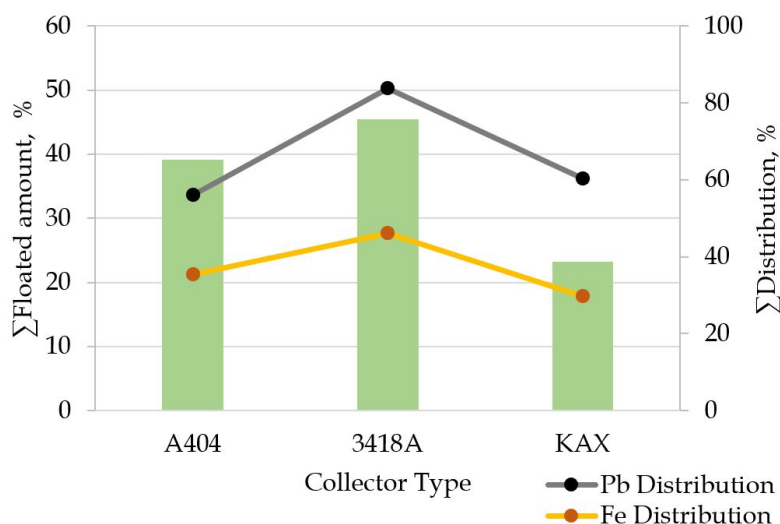


Fig. 4. Effect of collector type on Pb flotation performance of the -0.1 mm flotation feed at natural pH

As shown in Fig. 4, collector type strongly influenced Pb flotation performance, particularly Pb recovery, floated amount, Fe co-recovery, and residual Pb content in the tailings. Among the tested collectors, 3418A provided the highest cumulative Pb recovery, whereas KAX resulted in the lowest floated amount and relatively lower Fe recovery.

In the KAX test, the cumulative floated products represented 23.3% of the feed mass and recovered 60.3% of Pb. The cumulative Pb grade of the floated products was approximately 8.7%, while 29.7% of Fe also reported to the floated products. The final tailing contained 1.74% Pb and accounted for 39.7% of the Pb distribution, indicating that a considerable portion of Pb remained unrecovered despite the relatively low mass pull.

A404 produced a higher floated amount, corresponding to 39.2% of the feed mass, but did not improve Pb recovery. The cumulative Pb recovery was 56.1%, with a floated Pb grade of approximately 7.2%, while 35.4% of Fe was recovered in the floated products. The final tailing contained 3.61% Pb and represented 43.9% of the Pb distribution. Therefore, A404 was less effective than KAX and 3418A under the applied conditions, particularly in terms of reducing Pb losses to the tailing.

Among the tested collectors, 3418A produced the highest cumulative Pb recovery, reaching 83.8%. In this test, the cumulative floated products represented 45.4% of the feed mass. The Pb content of the final tailing decreased to 0.95%, and only 16.2% of Pb remained in the non-floated product. However, this improvement was accompanied by a relatively high Fe recovery of 46.2% and a lower cumulative Pb grade of approximately 5.9%, mainly due to the increased mass pull and co-recovery of Fe-bearing particles. Therefore, although 3418A substantially improved Pb recovery, Pb-Fe selectivity remained limited. The calculated Pb/Fe selectivity ratios for the collector tests were 2.03, 1.58, and 1.81 for KAX, A404, and 3418A, respectively. These values were calculated as follows: $KAX = 60.3/29.7 = 2.03$, $A404 = 56.1/35.4 = 1.58$, and $3418A = 83.8/46.2 = 1.81$. Although KAX showed the highest Pb/Fe selectivity ratio, its Pb recovery was considerably lower than that obtained with 3418A. Therefore, 3418A was selected for the subsequent sulfidization-assisted flotation tests because it provided the highest Pb recovery and the lowest Pb content in the final tailing among the tested collectors. Therefore, Na_2S was introduced as a sulfidizing reagent to improve the flotation response of oxidized Pb-bearing phases by

forming sulfide-like surface species and promoting collector adsorption. However, the simultaneous recovery of Fe-bearing particles indicated that the collector alone was not sufficient to achieve effective Pb-Fe separation. Therefore, Na_2S was introduced as a sulfidizing reagent to improve the flotation response of oxidized Pb-bearing phases by forming sulfide-like surface species and promoting collector adsorption. Previous studies on oxidized Pb and Cu-Pb-Zn ores have similarly shown that the effectiveness of sulfidization-assisted flotation depends strongly on sulfide dosage, pulp pH, and collector type (Malghan, 1986; Xie et al., 2020).

Sulfidization tests were carried out using the -0.1 mm flotation feed at natural pH, approximately pH 10, with 3418A as the collector and MIBC as the frother. Na_2S was added at dosages of 500, 1000, and 1500 g/Mg to evaluate the effect of surface activation on Pb flotation from the Fe-rich lead-acid battery recycling slag. Fig. 5 presents the cumulative flotation results in terms of floated amount and the distributions of Pb, Fe, and S.

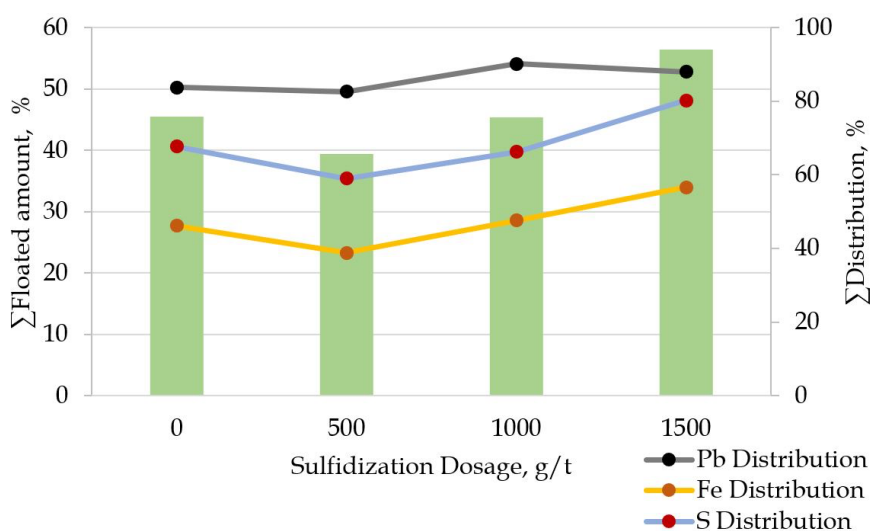


Fig. 5. Effect of sulfidization dosage on Pb flotation performance of the -0.1 mm flotation feed using 3418A as collector at natural pH

As shown in Fig. 5, Na_2S addition improved Pb flotation response, although the effect depended strongly on sulfidization dosage. At 500 g/Mg Na_2S , the cumulative floated products represented 39.4% of the feed mass and recovered 82.6% of Pb, while the final tailing contained 1.57% Pb and accounted for 17.4% of the Pb distribution. This indicates that partial activation of Pb-bearing surfaces was achieved; however, Pb losses to the tailing remained noticeable. In addition, 38.8% of Fe was reported to the floated products, indicating limited Pb-Fe selectivity.

Increasing the Na_2S dosage to 1000 g/Mg enhanced Pb recovery to 90.2%, with a cumulative floated amount of 45.4%. Under this condition, the final tailing contained only 0.91% Pb and accounted for 9.8% of the Pb distribution, representing the highest Pb recovery and the lowest Pb loss among the tested sulfidization conditions. However, Fe recovery also increased to 47.6%, showing that the improved Pb recovery was accompanied by additional recovery of Fe-bearing particles.

Further increasing the Na_2S dosage to 1500 g/Mg did not improve Pb recovery. Although the final tailing had the lowest Pb grade of 0.65%, the Pb distribution in the tailing increased to 12.0%, while the cumulative floated amount and Fe recovery increased to 56.4% and 56.6%, respectively. The S distribution in the floated products also reached 80.3%, suggesting that excessive sulfidization promoted non-selective flotation and concentrate dilution by non-target Fe- and S-bearing phases. The calculated Pb/Fe selectivity ratios for the sulfidization tests were 1.81, 2.13, 1.89, and 1.55 for 0, 500, 1000, and 1500 g/Mg Na_2S , respectively. These values were calculated as follows: 0 g/Mg Na_2S = $83.8/46.2 = 1.81$, 500 g/Mg Na_2S = $82.6/38.8 = 2.13$, 1000 g/Mg Na_2S = $90.2/47.6 = 1.89$, and 1500 g/Mg Na_2S = $88.0/56.6 = 1.55$. Although 500 g/Mg Na_2S provided the highest Pb/Fe selectivity ratio, 1000 g/Mg Na_2S resulted in the highest Pb distribution to the floated products and the lowest Pb distribution

in the tailing. Therefore, 1000 g/Mg Na₂S was selected as the most favorable condition considering both Pb recovery and residual Pb losses.

The improvement in Pb flotation after Na₂S addition can be attributed to the formation of sulfide-like surface species on oxidized Pb-bearing particles, which enhances collector adsorption. However, excessive sulfide addition may reduce selectivity by promoting non-selective flotation or altering reagent adsorption conditions. Therefore, careful control of Na₂S dosage is essential for improving Pb recovery while limiting Fe co-recovery, consistent with previous studies on sulfidization-assisted flotation of oxidized Pb and Cu-Pb-Zn ores (Malghan, 1986; Xie et al., 2020).

4. Conclusions

This study investigated Pb recovery from lead-acid battery recycling slag using physical pre-concentration and sulfidization-assisted flotation. The slag was characterized as an Fe-rich secondary resource containing 40.1% Fe and 4.3% Pb, with Pb broadly distributed across all particle size flotation feeds. Consequently, particle size classification alone was insufficient for selective Pb enrichment. Gravity separation provided only limited upgrading, producing heavy products containing 10.3–11.0% Pb, while low mass yields restricted Pb recovery. Similarly, magnetic separation successfully upgraded Fe-bearing phases but failed to selectively recover Pb because Pb was likely associated with magnetic Fe-bearing particles. These results show that conventional physical beneficiation methods alone are insufficient to recover Pb from this complex slag.

Flotation significantly improved Pb recovery. Although the –0.2 mm flotation feed achieved the highest Pb recovery during the preliminary particle-size tests, the –0.1 mm flotation feed was selected for reagent optimization as a finer feed to evaluate reagent effects under improved liberation conditions. Natural pH (approximately pH 10) was adopted as the operating condition because it provided a practical balance between Pb recovery, Pb-Fe selectivity, and reagent consumption. Among the tested collectors, 3418A produced the highest Pb recovery (83.8%). Sulfidization with Na₂S further enhanced flotation performance. A dosage of 1000 g/Mg increased Pb recovery to 90.2% while reducing Pb losses to the tailings to 9.8%. Higher Na₂S dosages did not improve performance and instead increased Fe recovery and mass pull, indicating reduced flotation selectivity.

Overall, the results demonstrate that sulfidization-assisted flotation is an effective approach for recovering Pb from Fe-rich lead-acid battery recycling slags. Although physical pre-concentration alone was insufficient, it provided valuable information for process design and, when combined with flotation, offers a promising route for the sustainable valorization of secondary Pb resources. Future studies should address both the fundamental mechanisms and process optimization of Pb recovery from lead-acid battery recycling slag. From a mechanistic perspective, further investigations are needed to clarify the surface interactions and adsorption behavior of collectors and sulfidizing reagents on Pb- and Fe-bearing slag phases. Detailed mineralogical characterization should also be undertaken to elucidate the phase associations responsible for the parallel behavior of Pb and Fe observed during the separation tests. In particular, quantitative phase analysis, including determination of the X-ray amorphous fraction, combined with microstructural and elemental characterization, would help determine whether Pb and Fe occur within common phases or as closely associated constituents in the slag matrix. From an application-oriented perspective, design of experiments (DOE) could be employed to systematically quantify the individual and interactive effects of key flotation variables, including particle size, pH, collector dosage, sulfidization dosage, and conditioning time, and thereby identify more selective operating conditions. Integrated flowsheets combining flotation with physical separation techniques should also be investigated to improve Pb-Fe selectivity while minimizing reagent consumption and operating costs. These studies would provide a stronger basis for understanding the limited Pb-Fe selectivity observed in the present work and for developing more efficient beneficiation strategies.

Acknowledgments

This study was carried out within the scope of the TÜBİTAK 2209-A University Students Research Projects Support Program. The authors gratefully acknowledge TÜBİTAK for its financial support. A part of the results reported in this paper were presented at XX Balkan Mineral Processing Congress

BMPC 2026 entitled “Research on the recovery of lead and iron from the slag of a lead-acid battery recycling plant using physical methods”.

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