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Antimony Hydrometallurgy from Primary and Secondary Sources (2020–2025): Critical Review of Leaching Systems and Downstream Separation

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ABSTRACT

Original Research Article

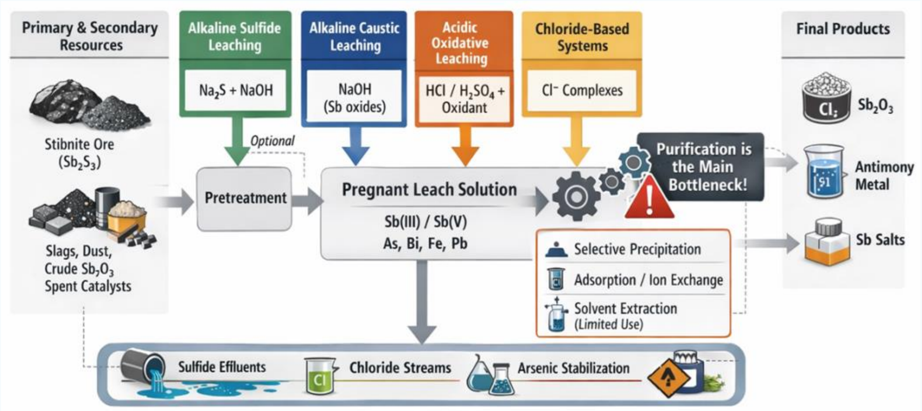
Antimony (Sb) is a critical element with growing strategic importance, yet its hydrometallurgical processing remains challenged by complex mineralogy, impurity-rich feeds, and limited industrial validation. This review critically examines recent advances in pretreatment, leaching, purification, and final recovery strategies for Sb-bearing materials, with emphasis on their integration at the flowsheet level. Pretreatment approaches primarily condition mineralogical accessibility rather than directly enhance Sb recovery, while diverse leaching systems consistently achieve high extraction efficiencies but generate chemically complex pregnant leach solutions. Across the literature, downstream purification is identified as the principal bottleneck, driven by impurity co-dissolution, selectivity limitations, and solution stability. Despite extensive laboratory-scale investigations, many separation strategies remain weakly validated with respect to reagent regeneration, long-term operability, and compatibility with final recovery routes. This review highlights that technical success at the unit-operation level does not necessarily translate into industrial feasibility, underscoring the need for integrated flowsheet validation, impurity-aware design, and technology readiness assessment. Future research should prioritize holistic process integration and scale-relevant evaluation to bridge the gap between academic development and sustainable antimony production.

Keywords: Antimony, Hydrometallurgy, Stibnite, Secondary resources, Alkaline sulfide leaching, Solution purification, Impurity control, Circular economy.

Highlights

- Pretreatment primarily conditions mineralogical accessibility rather than directly enhancing Sb recovery.
- High Sb extraction efficiencies during leaching frequently generate impurity-rich and unstable solutions.
- Purification and separation represent the principal technical and economic bottlenecks in Sb hydrometallurgy.
- Many proposed separation strategies remain weakly validated at the integrated flowsheet level.
- Bridging unit-operation studies with technology readiness assessment is essential for industrial deployment.

Graphical Abstract



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1. Introduction

Antimony (Sb) has re-emerged as a strategically important metalloid for modern industry, underpinning applications ranging from flame retardants and lead–acid battery alloys to electronics, specialty catalysts, and emerging functional materials. At the same time, Sb is widely recognized for its environmental persistence and potential toxicity when released from ores, mine wastes, and metallurgical residues, which has intensified regulatory scrutiny across mining and metallurgical value chains (Tylenda et al., 2022). From a supply-chain perspective, the high geographical concentration of primary Sb production and the growing complexity of global trade networks have further reinforced concerns about long-term availability, price volatility, and strategic dependence (Zhao et al., 2023). These drivers have collectively renewed interest in alternative processing routes and in the valorization of secondary antimony-bearing resources as part of broader circular-economy strategies.

Geochemically and metallurgically, antimony occurs across a wide range of primary and secondary feedstocks, whose heterogeneity poses a fundamental challenge to process design. Primary resources are dominated by stibnite (Sb_2S_3), often associated with gold, arsenic, and other chalcophile elements, whereas secondary resources include flotation tailings, smelter slags and dusts, anode slimes, crude antimony oxides, spent catalysts, and industrial side streams generated during copper and lead refining (Ling et al., 2021). In both contexts, the strong coupling between Sb and elements such as As, Bi, Fe, and Pb governs dissolution behavior, solution chemistry, and ultimately product quality, making simplified or universal flowsheets unreliable (Yu et al., 2023). In parallel, environmental studies have highlighted the mobility and long-term persistence of Sb in mine wastes and contaminated soils, underscoring the need for processing strategies that control not only metal recovery but also residue stability and effluent quality (Drahota et al., 2023; Zhou et al., 2024).

Within this framework, hydrometallurgical processing has gained renewed momentum between 2020 and 2025 as a flexible, potentially lower-impact alternative or complement to conventional pyrometallurgical routes. Recent studies have explored a range of leaching chemistries, including alkaline sulfide and caustic systems for sulfide-rich or oxide feeds, oxidative acidic leaching for oxidized materials, and chloride-based systems for complex secondary streams and industrial side solutions (Dembele et al., 2022). For stibnite and related tailings, alkaline hydrometallurgical routes remain attractive for their inherent selectivity toward Sb and compatibility with residue reprocessing at semi-pilot and scale-up levels (Dembele et al., 2024; Dembele et al., 2025). Complementary process intensification strategies, such as microwave-assisted heating integrated with alkaline sulfide solutions, have been proposed to improve kinetics and reduce energy and reagent consumption in Sb-bearing copper concentrates (Luo et al., 2024). Chloride-based media, in turn, have been increasingly applied to copper-industry side streams where antimony appears as an impurity or recoverable by-product, requiring careful redox control and selective hydrolysis to manage co-dissolved bismuth and arsenic (Benabdallah et al., 2023).

Beyond conventional chemical leaching, biohydrometallurgical approaches and electrochemical methods have also been reported for antimony-bearing materials. Bioleaching and microbial oxidation studies provide valuable mechanistic insight into Sb dissolution pathways under ambient conditions, yet their technological maturity and scalability remain limited compared with chemical hydrometallurgical systems (Aghazadeh et al., 2023). Electrochemical techniques, including electrodeposition and membrane-assisted processes, have shown promise as downstream recovery or polishing steps for Sb-containing effluents, particularly in copper electrorefining circuits, though their applicability is strongly constrained by solution composition and mass-transfer control (Hernández-Pérez et al., 2023).

A critical assessment of the 2020–2025 literature, however, shows that leaching efficiency alone is a weak indicator of overall process viability. Across primary and secondary feedstocks, downstream purification of pregnant leach solutions consistently emerges as the dominant technical and economic bottleneck, particularly for separating antimony from arsenic, bismuth, iron, and lead (Ling et al., 2022). Small changes in pH, redox potential, or ligand chemistry can trigger hydrolysis, co-precipitation, or impurity recycling, undermining closed-loop operation and product specifications, especially in chloride- and sulfur-rich systems (Díaz-Gutiérrez et al., 2025). Consequently, recent research has increasingly focused on integrated purification strategies that combine selective precipitation or hydrolysis with solvent extraction, adsorption, or ion-exchange steps to deliver high-purity Sb_2O_3 or antimony metal while minimizing secondary waste generation (Benabdallah et al., 2023; Ibrahim et al., 2025). In parallel, a smaller but growing body of work has proposed closed-loop hydrometallurgical concepts explicitly targeting solution regeneration, residue stabilization, and long-term operational robustness rather than single-pass extraction yields (Eshtiaghi & Tabaian, 2025).

In this context, the objective of this critical review is to synthesize and evaluate advances in hydrometallurgical antimony recovery from primary ores and secondary resources published between 2020 and 2025. The review focuses on (i) feedstock-driven selection of leaching chemistry, including alkaline sulfide/caustic, acidic oxidative, and chloride-based systems; (ii) the role of antimony speciation and redox control in governing selectivity and impurity behavior; (iii) purification and separation strategies for As-, Bi-, Fe-, and Pb-rich solutions, which constitute the principal flowsheet bottleneck; and (iv) environmental performance, effluent management, and indicators of scale-up and industrial readiness. Rather than compiling extraction yields in isolation, the review emphasizes reagent consumption, impurity trajectories, solution recycle behavior, and product quality constraints to enable meaningful cross-study comparison. The following section describes the methodology for literature selection, screening, and critical analysis, outlining the databases consulted, search strategies, inclusion and exclusion criteria, and quality-assessment framework used to construct the evidence base for this review.

2. Methodology

This critical review was conducted following a structured literature survey and screening strategy adapted from the PRISMA (Preferred Reporting Items for Systematic Reviews and Meta-Analyses) framework to ensure transparency, reproducibility, and methodological rigor (Page et al., 2021). Peer-reviewed publications published between January 2020 and December 2025 were identified using major scientific databases, including Scopus, Web of Science, Science Direct, and Google Scholar. These searches were complemented by targeted screening of leading journals in hydrometallurgy, mineral processing, and environmental metallurgy.

Search strings combined core terms related to antimony and hydrometallurgical processing (e.g., *antimony*, *Sb*, *hydrometallurgy*, *leaching*, *purification*, *secondary resources*) with additional keywords tailored to specific feedstocks, leaching chemistries, and separation techniques. This strategy ensured broad coverage of both conventional and emerging processing routes while maintaining relevance to applied hydrometallurgical flowsheets.

After removing duplicates, titles and abstracts were screened to exclude studies outside the scope of antimony recovery, purely geochemical or toxicological investigations without process relevance, and publications lacking experimental, mechanistic, or techno-economic information. Full-text assessment was then conducted to retain studies reporting explicit processing conditions, including leaching media, operating parameters, impurity behavior, and downstream purification or recovery steps. Particular emphasis was placed on contributions addressing secondary antimony-bearing resources, solution purification strategies, and closed-loop or scale-up considerations.

Based on these criteria, 99 publications were selected and critically analyzed in this review. To support a critical rather than purely descriptive assessment, the selected studies were categorized by (i) feedstock type (primary ores versus secondary resources), (ii) leaching chemistry (alkaline sulfide/caustic, acidic oxidative, or chloride-based systems), and (iii) downstream purification strategy (hydrolysis or selective precipitation, adsorption or ion exchange, solvent extraction, and electrochemical recovery). The comparative analysis focused on reported extraction efficiency, reagent consumption, impurity separation performance, environmental implications, and indicators of technological maturity.

To ensure transparency and reproducibility in the literature selection process, the sequential steps of identification, screening, eligibility assessment, and final inclusion of studies are summarized in Figure 1, following the PRISMA framework.

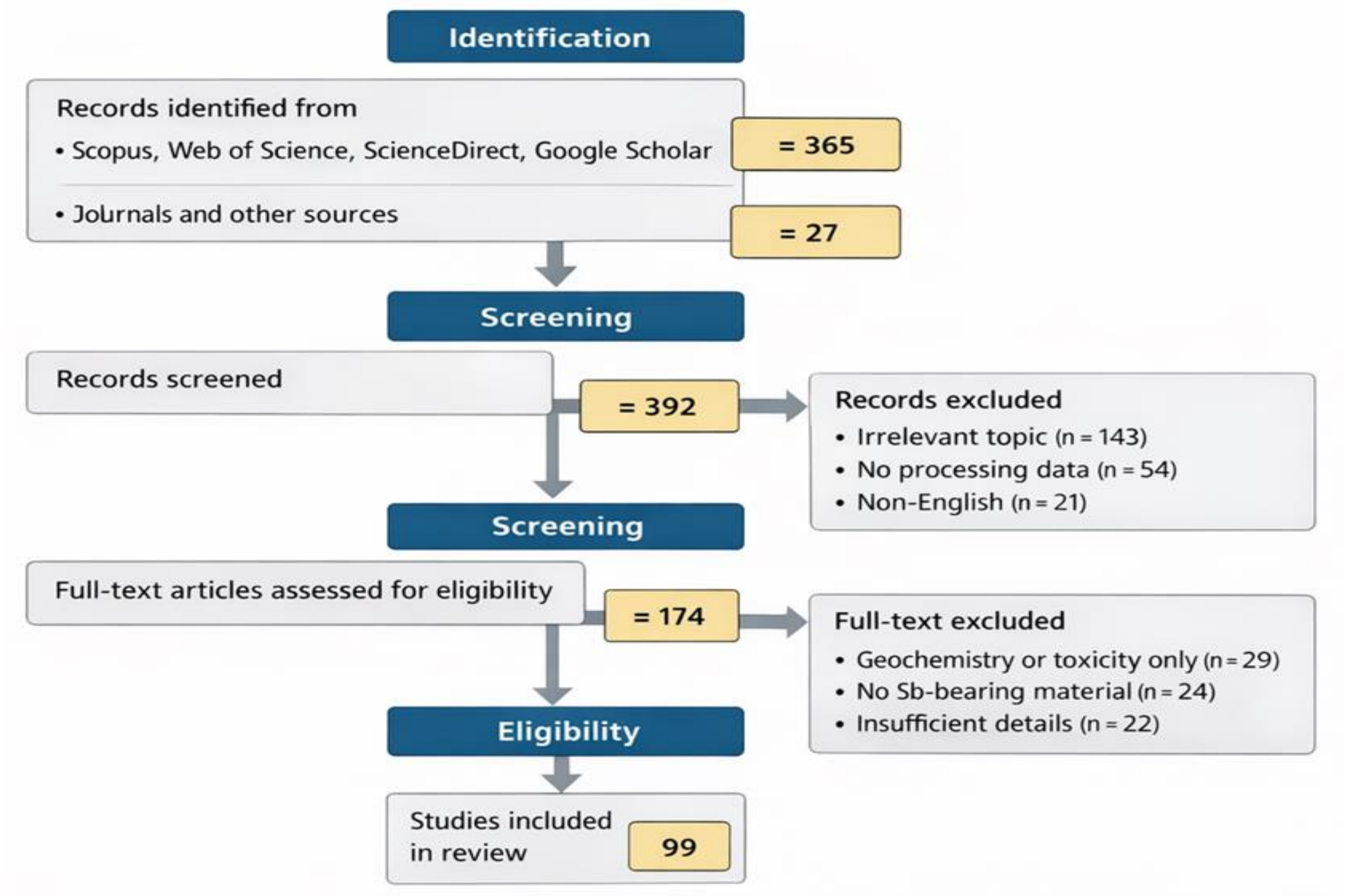

Figure 1. PRISMA flowchart summarizing the literature search, screening, eligibility assessment, and selection process applied in this critical review. Adapted from Page et al. (2021).

As shown in Figure 1, an initial set of records was identified through database searches and complementary sources. After duplicate removal and title–abstract screening, studies not directly related to antimony recovery, lacking processing data, or outside the defined scope were excluded. Full-text evaluation further refined the dataset by removing contributions focused exclusively on geochemistry or toxicity, or those reporting insufficient experimental detail. The final dataset comprised 99 peer-reviewed publications, forming the basis for the critical comparative analysis presented in this review.

Although this review focuses on hydrometallurgical studies published between 2020 and 2025, the analytical framework adopted here builds on a well-established understanding of antimony mineralogy, metallurgy, and environmental behavior developed over several decades. Foundational contributions on the historical evolution of antimony extraction and use (Dillis & Degryse, 2020), global production and resource context (He et al., 2021; Motang & Kuangdi, 2023), and toxicological and environmental considerations (Tylenda et al., 2022) provide essential background for interpreting recent process-oriented advances within broader technological and societal contexts.

3. Feedstocks and Mineralogy-Controlled Behavior

Hydrometallurgical recovery of antimony is governed not only by reagent chemistry but, more fundamentally, by the feedstock's mineralogical and chemical characteristics. Recent studies reveal recurring patterns showing that dissolution efficiency alone is a poor predictor of overall process performance, because impurity mobilization and mineralogical barriers often dominate downstream separation complexity and operational robustness.

Figures 2–4 collectively synthesize these constraints. Figure 2 establishes the link between dominant Sb-bearing mineral phases and preferred hydrometallurgical routes; Figure 3 integrates feedstock chemical signatures with leaching–purification logic for both primary ores and secondary resources; and Figure 4 illustrates the mineralogical mechanisms that limit antimony leaching at the particle scale.

The strong dependence of antimony leaching behavior on feedstock mineralogy and phase associations is summarized schematically in Figure 2, which links dominant mineral forms to preferred leaching routes, impurity trajectories, and downstream processing constraints.

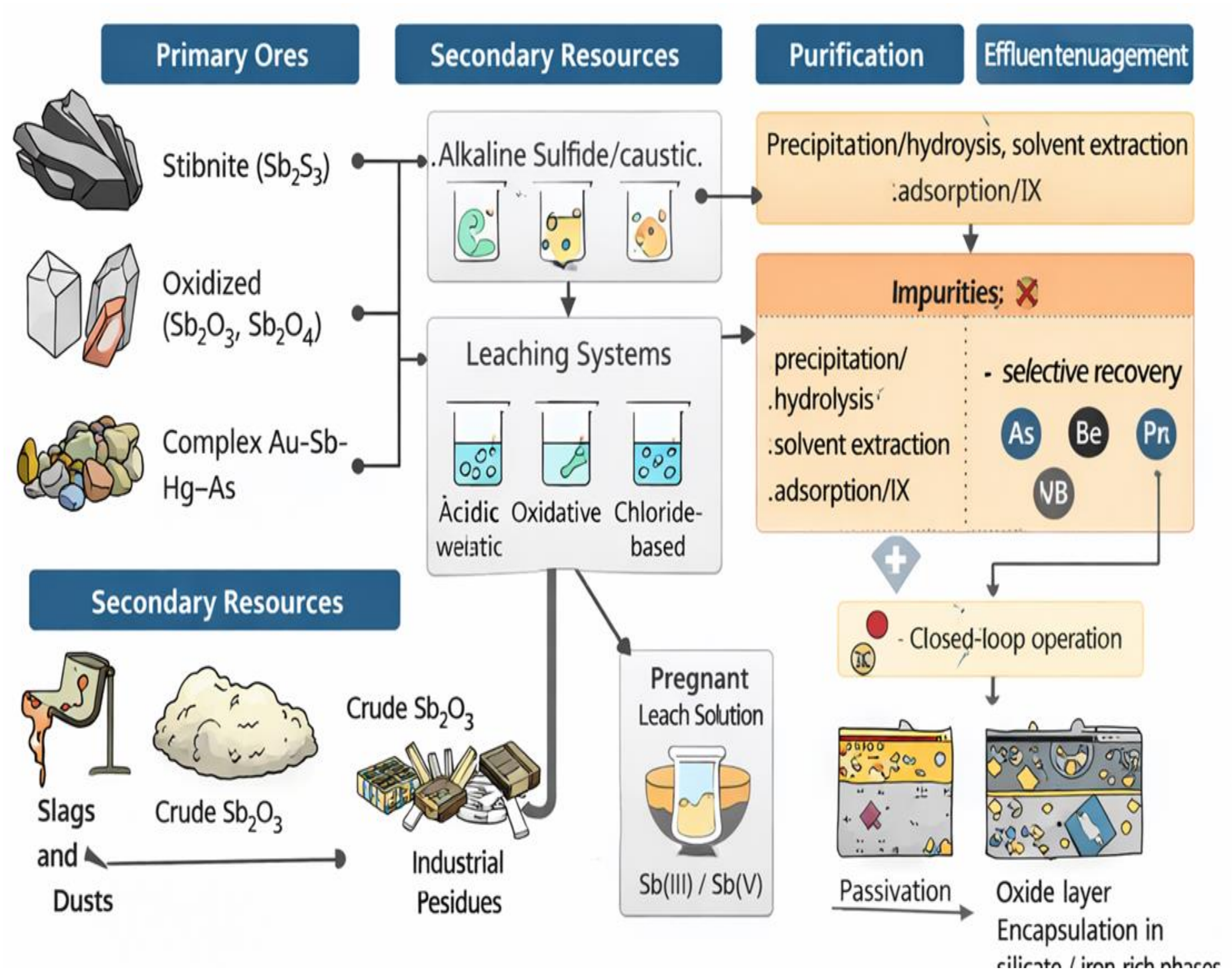

Figure 2. Conceptual mineralogy–process interaction scheme for hydrometallurgical antimony recovery from primary ores and secondary resources. Adapted from Majzlan (2021).

As shown in Figure 2, variations in mineralogical binding and matrix association produce fundamentally different leaching responses and purification challenges. While sulfide-dominated ores may favor alkaline sulfide systems, oxidized and secondary materials often require more aggressive or chemically complex leaching conditions. Across all feedstocks, the scheme underscores that downstream purification—rather than dissolution itself—governs process robustness, environmental performance, and product quality.

3.1. Primary Antimony Ores

Primary antimony ores are dominated by stibnite (Sb_2S_3), which commonly occurs as a relatively pure sulfide or in association with gold and other chalcophile elements, forming complex Sb–Au, Sb–Hg, or Sb–As mineral systems. In these ores, antimony is structurally bound within sulfide lattices, yielding high theoretical recoverability but also pronounced sensitivity to surface oxidation and sulfur chemistry during leaching (Dembele et al., 2022).

Oxidized antimony ores, containing phases such as senarmontite or valentinite (Sb_2O_3) and mixed Sb_2O_4 species, exhibit markedly different dissolution behavior, often favoring alkaline caustic or acidic oxidative leaching routes depending on impurity association and gangue mineralogy (Zekavat et al., 2021).

Complex Sb–Au ores are particularly challenging because antimony often acts as a penalty element in gold processing circuits. In these systems, Sb-bearing minerals may coexist with or encapsulate gold particles, complicating flotation and downstream hydrometallurgical treatment (Smith et al., 2022). From a hydrometallurgical perspective, the presence of arsenic-bearing phases alongside stibnite further constrains selectivity, as Sb and As often overlap in their dissolution windows and exhibit competing hydrolysis behavior (Zhang et al., 2023).

To facilitate comparison across recent studies, Table 1 summarizes the primary antimony ore classes reported from 2020 to 2025, highlighting dominant Sb-bearing phases, typical associated elements, and the hydrometallurgical leaching systems most frequently investigated for each ore type.

Table 1. Classification of primary antimony ores, associated elements, and preferred hydrometallurgical leaching routes. Adapted from Majzlan (2021) and Dembele et al. (2022)

Primary ore type	Dominant Sb-bearing phase(s)	Typical associated elements	Mineralogical implications	Preferred hydrometallurgical routes (2020–2025)	Key constraints
Stibnite-rich ores	Sb_2S_3 (stibnite)	As, Fe, Pb, Au	Sulfide lattice; prone to surface oxidation and sulfur passivation	Alkaline sulfide leaching (Na_2S–NaOH); alkaline caustic leaching after pretreatment	Elemental sulfur passivation; Asco-dissolution
Oxidized antimony ores	Sb_2O_3 (senarmontite, valentinite), Sb_2O_4	Fe, Pb, SiO_2	Oxide phases with variable solubility; limited sulfur interference	Alkaline caustic leaching (NaOH); acidic oxidative leaching (HCl/H_2SO_4 + oxidant)	Encapsulation in iron oxides; hydrolysis control
Complex Sb–Au ores	Sb_2S_3 ± Au-bearing sulfides	Au, As, Fe, Pb	Fine intergrowths; Sb often acts as a penalty element	Selective alkaline sulfide leaching prior to Au recovery	Overlapping Sb–As chemistry; gold deportment
Sb–Hg ores	Sb_2S_3 ± Hg sulfides	Hg, As	Volatile and environmentally sensitive phases	Controlled alkaline sulfide leaching; staged purification	Hg stabilization; effluent management
Sb–As dominant ores	Mixed Sb–As sulfides/oxides	As, Fe	Strong chemical affinity between Sb and As	Alkaline sulfide or acidic oxidative leaching with selective purification	Sb–As separation during purification

As shown in Table 1, the mineralogical form of antimony exerts first-order control over both leaching response and downstream process constraints. Sulfide-dominated ores, particularly stibnite-rich systems, are generally compatible with alkaline sulfide leaching but remain highly sensitive to surface oxidation and sulfur passivation. In contrast, oxidized antimony ores tend to favor alkaline caustic or acidic oxidative routes, although encapsulation within iron oxides or silicate gangue can significantly limit dissolution efficiency.

Complex Sb–Au, Sb–Hg, and Sb–As ores are the most challenging feedstocks, in which overlapping dissolution behavior and impurity co-mobilization shift the main process bottleneck from leaching to purification and effluent management. These observations reinforce the need to evaluate hydrometallurgical routes in conjunction with mineralogical constraints rather than on extraction yields alone.

3.2. Secondary Resources and Typical "Chemical Signatures."

Secondary antimony-bearing resources encompass a broad range of materials generated during mining, smelting, and refining operations, including flotation tailings, smelter slags and dusts, anode slimes, crude antimony oxides, and industrial by-products from copper and lead metallurgy. These resources span a wide spectrum, ranging from industrially well-defined residues (such as electrorefining slimes and electrolytes) to highly heterogeneous materials including electronic waste and thermally transformed calcines. Across this spectrum, antimony recovery is frequently constrained not by dissolution efficiency but by multi-metal solution management and Sb–As coupling, which govern selectivity and downstream purification rather than extraction alone (Barragan et al., 2020; Brožová et al., 2021; Xue et al., 2021; Xiao et al., 2023; Tang et al., 2022; Rakhimov et al., 2024).

In contrast to primary ores, secondary resources are typically characterized less by discrete mineral phases and more by heterogeneous chemical "signatures" that reflect their thermal history, chemical conditioning, and metallurgical processing routes (Ling et al., 2021). Crude Sb_2O_3, for instance, may contain significant levels of arsenic, lead, iron, and silica, while smelter slags often host antimony within glassy silicate matrices or as finely dispersed oxide phases, strongly limiting direct leachability (Yu et al., 2023).

Recent studies consistently emphasize that detailed chemical and mineralogical characterization of secondary resources is a decisive prerequisite for defining viable valorization routes, as antimony is rarely present as a free, readily leachable phase (Ling et al., 2024). Consequently, secondary feedstocks often require more aggressive or tailored hydrometallurgical systems—such as chloride-based leaching, oxidative conditioning, or integrated roasting–leaching schemes—to overcome matrix effects and impurity constraints, shifting the process bottleneck upstream to feed conditioning and downstream to purification (Alguacil, 2025).

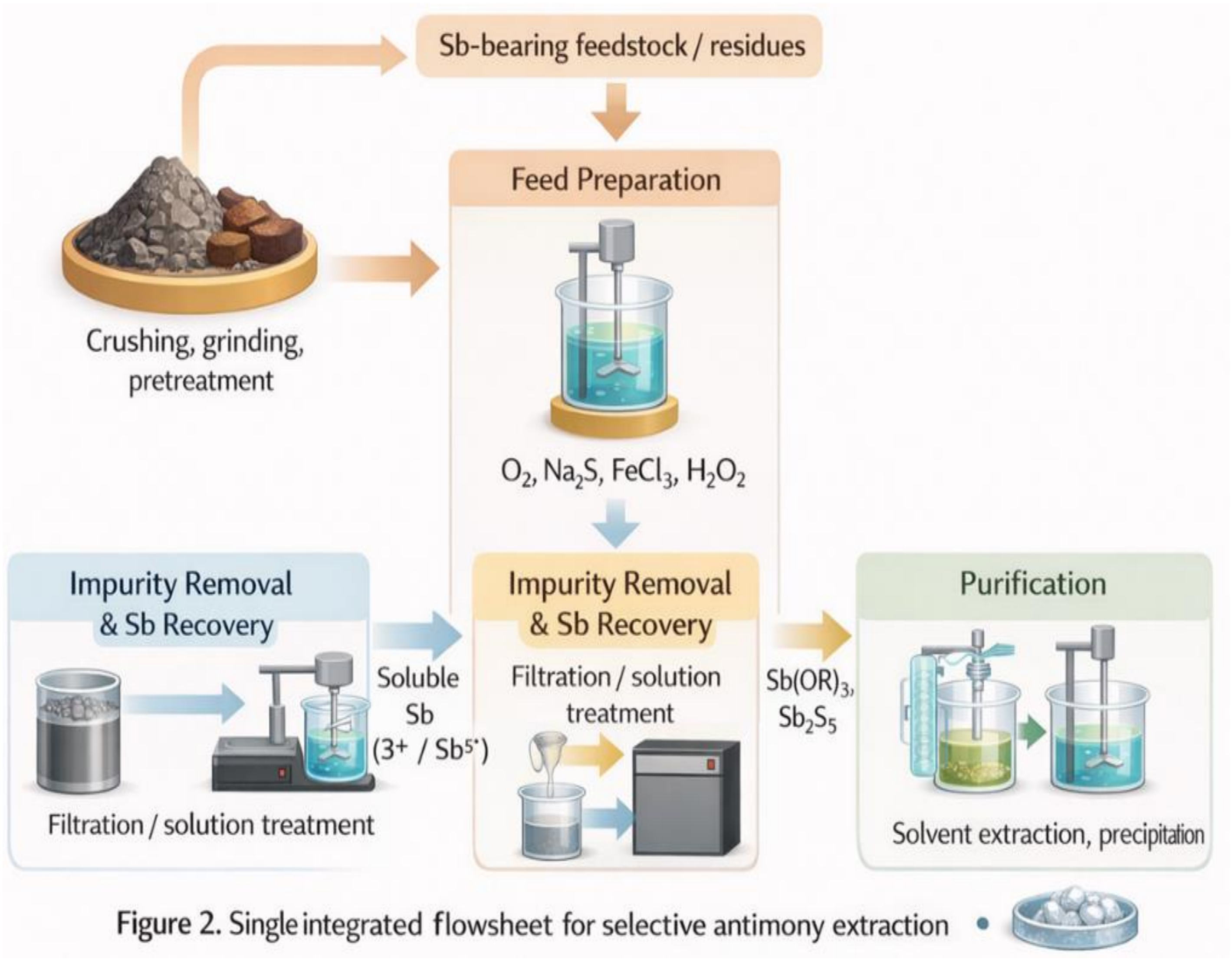

Figure 3. Single integrated conceptual scheme linking Sb-bearing feedstock characteristics to hydrometallurgical processing and purification. Adapted from: Dembele et al. (2022) and Alguacil (2025).

As shown in Figure 3, variations in feed composition and chemical signatures create distinct leaching and purification challenges, even when similar hydrometallurgical reagents are used. Although Sb dissolution can be readily achieved under alkaline, oxidative, or chloride-based conditions, the co-dissolution of impurities such as As, Fe, and Pb often determines the complexity of subsequent separation steps. Accordingly, process selectivity and robustness are governed not only by dissolution efficiency but also by the compatibility between feed chemistry and purification strategy.

3.3. Mineralogical Control of Leaching Behavior

Across both primary and secondary feedstocks, mineralogical factors exert first-order control on antimony leaching behavior. Passivation phenomena are frequently reported during alkaline sulfide or caustic leaching of stibnite, where the formation of elemental sulfur or secondary antimony oxides progressively inhibits dissolution (Moosavi Nejad, 2020).

In oxidized or thermally treated materials, antimony may be encapsulated in iron oxides or ferrites, limiting lixiviants' access and requiring either reductive pretreatment or intensified leaching conditions (Zhang et al., 2021). Encapsulation in silicate or glassy matrices is particularly problematic in smelter slags and dusts, where antimony is distributed throughout chemically resistant networks (Ling et al., 2021).

These mineralogical constraints help explain why high laboratory leaching yields reported for simplified systems often fail to translate into robust industrial performance when applied to real-world, heterogeneous feedstocks. In such cases, leaching efficiency depends on matrix breakdown rather than intrinsic Sb chemistry, increasing reagent consumption and waste generation.

Figure 4 further illustrates why these challenges persist even under aggressive leaching conditions, highlighting mineralogical barriers such as surface passivation, oxide-layer formation, and encapsulation within silicate or iron-rich matrices.

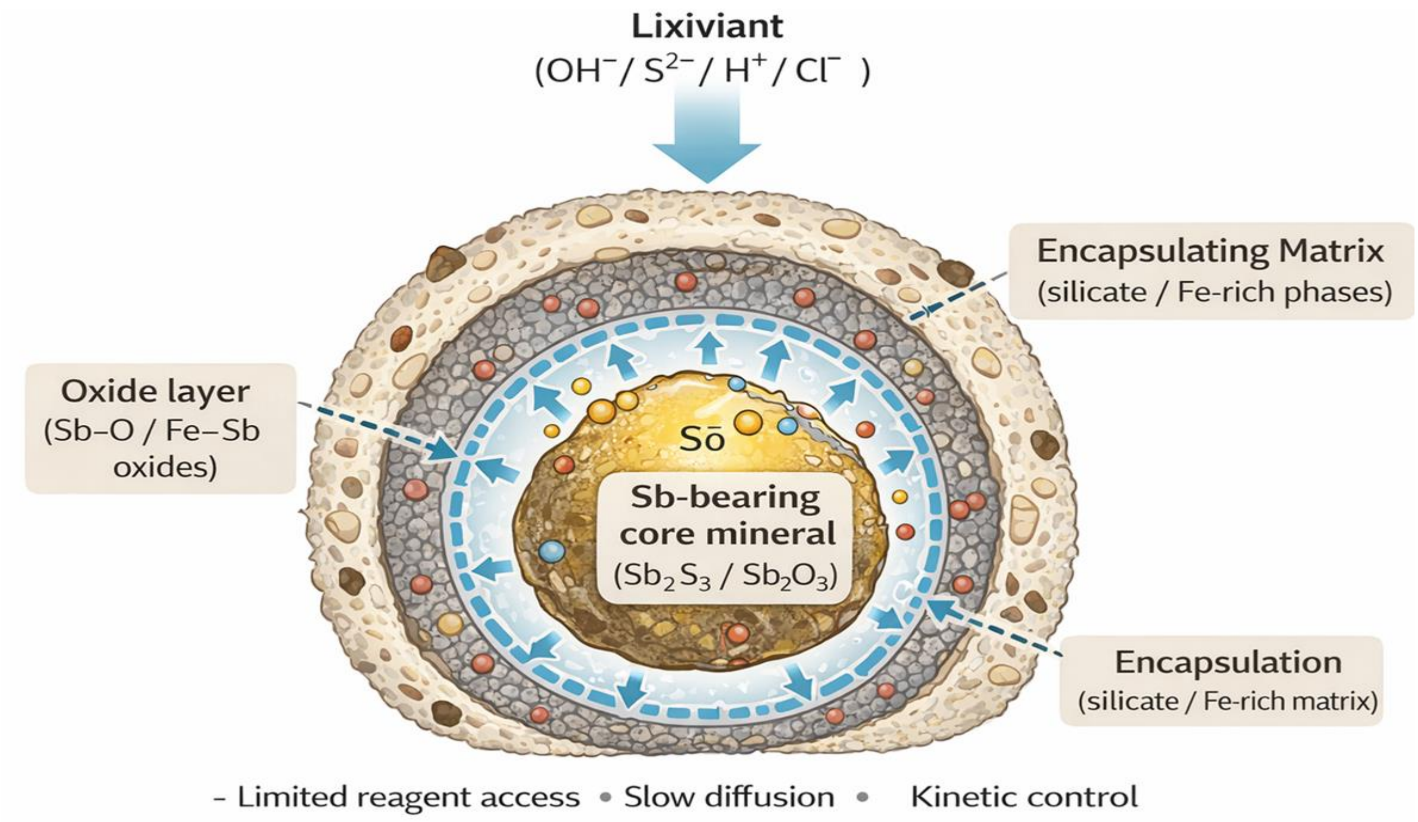

Figure 4. Single integrated conceptual diagram of mineralogical barriers controlling antimony leaching. Adapted from: Dembele et al. (2022) and Ling et al. (2024).

As summarized in Figure 4, Sb dissolution can be simultaneously hindered by (i) surface passivation (e.g., elemental sulfur and secondary oxides), (ii) formation of compact oxide layers that restrict mass transfer, and (iii) encapsulation of Sb-bearing phases within silicate or Fe-rich matrices. The coexistence of these barriers shifts the primary performance determinant from dissolution to pretreatment strategy, Eh–pH control, and purification design capable of handling co-dissolved impurities while maintaining product quality and operational stability (Alguacil, 2025; Ling et al., 2024).

Overall, the literature clearly shows that feedstock mineralogy governs not only the choice of hydrometallurgical leaching chemistry but also the feasibility of downstream purification and closed-loop operation. Failure to account for mineralogical controls—particularly passivation mechanisms and matrix encapsulation—has repeatedly led to overestimation of process performance at the laboratory scale. These observations underscore the need to evaluate leaching strategies in conjunction with impurity behavior and solution chemistry, as examined in detail in the following section on leaching systems and reaction mechanisms.

4. Pretreatment Strategies: The Decisive Step in Antimony Hydrometallurgy

Building directly on the mineralogical constraints discussed in Section 3, it is evident that pretreatment strategies—rather than leaching chemistry alone—frequently determine the technical feasibility, selectivity, and environmental robustness of antimony hydrometallurgical flowsheets. Although numerous studies report high Sb dissolution efficiencies under optimized leaching conditions, a critical examination of the 2020–2025 literature shows that the principal process bottlenecks are often upstream, at the level of feedstock conditioning and impurity control, rather than within the leaching step itself.

Across both primary ores and secondary Sb-bearing residues, pretreatment serves three distinct yet interrelated functions: (i) modifying Sb mineralogy and oxidation state to enable chemically compatible leaching routes, (ii) selectively removing or stabilizing deleterious impurities—most notably arsenic and bismuth—that otherwise compromise downstream selectivity, and (iii) enhancing reaction kinetics through particle activation, surface renewal, and mitigation of diffusion barriers. Collectively, these functions exert first-order control over leaching system selection and largely dictate the complexity, cost, and sustainability of subsequent purification stages.

4.1. Oxidative Pretreatment: From Sulfide to Oxide-Controlled Systems

Oxidative pretreatment—implemented via roasting, controlled oxidative conditioning, or chemical oxidation—has long been used to convert stibnite (Sb_2S_3) into oxide phases such as Sb_2O_3 and Sb_2O_4, enabling subsequent leaching in acidic or caustic alkaline media. Recent studies confirm that this step remains essential for processing sulfide-dominated primary ores and refractory concentrates, particularly when direct alkaline sulfide leaching is kinetically constrained or operationally undesirable (Li et al., 2020; Li et al., 2021; Rusalev et al., 2023).

At the same time, the literature highlights a critical trade-off with oxidative pretreatment. While oxidation generally improves antimony accessibility and broadens the range of applicable leaching systems, it may also promote the formation of dense oxide layers, ferrite phases, or complex mixed oxides that restrict mass transfer during leaching (Ling et al., 2021; Ling et al., 2024). In secondary resources—such as smelter slags, calcines, and metallurgical residues—excessive or poorly controlled oxidation can further entrap antimony within silicate or iron-rich matrices, ultimately reducing effective recoverability despite high nominal oxidation degrees (Yu et al., 2023; Alguacil, 2025).

Consequently, oxidative pretreatment plays a decisive role in defining the hydrometallurgical processing window for antimony-bearing materials by directly altering the dominant Sb mineral phases and their reactivity toward subsequent leaching systems. Across recent studies, oxidative conditioning has consistently enabled a strategic transition from sulfide-based alkaline leaching toward oxide-compatible acidic or oxidative routes. Figure 5 conceptually illustrates how this mineralogical transformation governs leaching pathway selection, impurity mobilization, and downstream purification requirements, reinforcing pretreatment as a critical route-defining decision rather than a secondary auxiliary step.

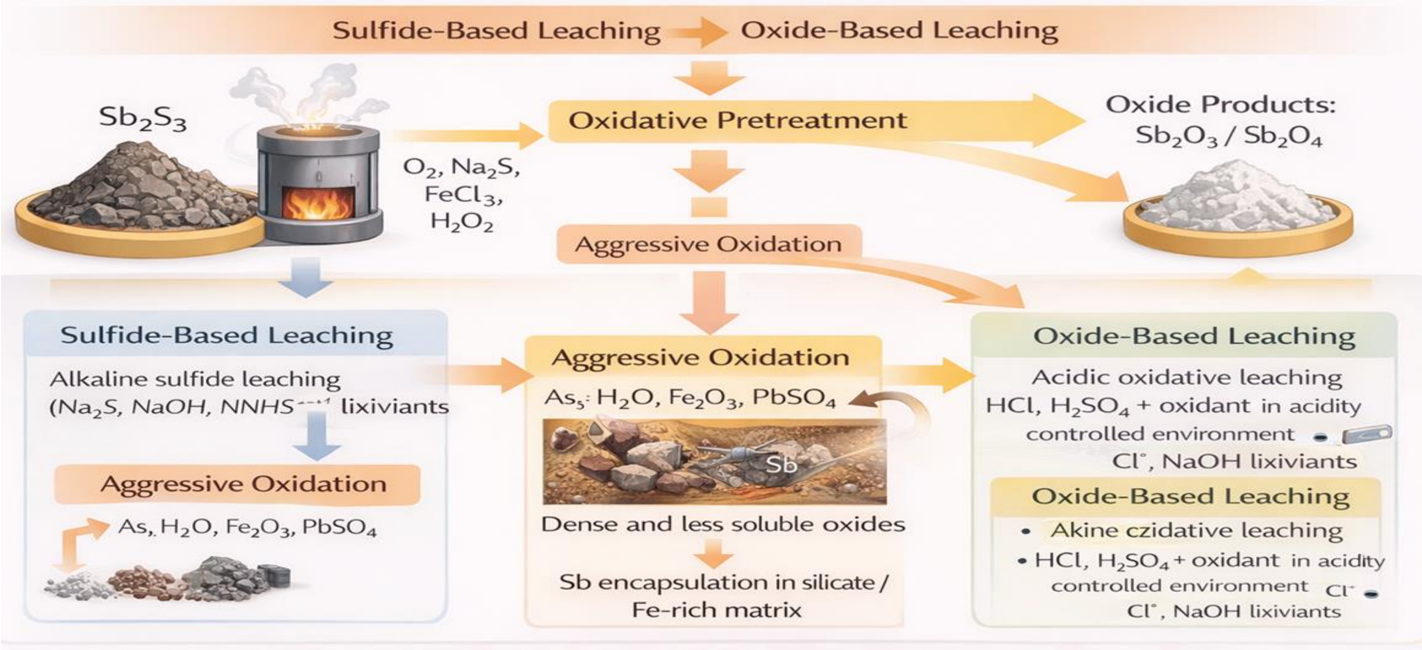

Figure 5. Conceptual role of oxidative pretreatment in controlling antimony mineralogy and leaching pathway selection. Adapted from Li et al. (2020), Ling et al. (2021), and Alguacil (2025).

As shown in Figure 5, oxidative pretreatment plays a dual role by enhancing Sb liberation while introducing new mineralogical barriers. The balance between sufficient sulfide oxidation and avoidance of excessive oxide encapsulation is a key design criterion, particularly when downstream purification selectivity is required.

4.2. De-arsenization and De-bismuthization: Pretreatment for “Clean” Antimony

When the primary objective is the production of high-purity antimony or Sb_2O_3, pretreatment strategies increasingly focus on the selective removal or stabilization of arsenic and bismuth, either prior to or deliberately integrated with, the leaching stage. This requirement is particularly acute for complex Sb–As ores, copper electrorefining residues, and crude antimony oxides, where As and Bi are critical penalty elements that compromise product quality and downstream process stability (Ling et al., 2022; Díaz et al., 2023; Díaz-Gutiérrez et al., 2025).

A recurring finding in recent studies is that arsenic and antimony frequently share overlapping dissolution windows under both alkaline and acidic leaching conditions, making direct leaching intrinsically non-selective (Wang et al., 2021; Zhang et al., 2023). Consequently, attempts to maximize Sb extraction without prior impurity control often yield complex pregnant solutions that require extensive purification, undermining process economics and environmental performance. This challenge is further exacerbated in secondary feedstocks, where Sb, As, and Bi are commonly co-distributed within fine-grained or amorphous matrices.

In response, pretreatment methods like controlled oxidation, selective hydrolysis, redox adjustment, and staged precipitation have become key operations, not just ancillary steps. These approaches are designed to separate Sb dissolution from the mobilization of As and Bi by stabilizing impurities in insoluble forms or removing them selectively before bulk leaching (Benabdallah et al., 2023; Luo et al., 2024). Notably, recent studies increasingly emphasize that managing As and Bi effectively should be integrated into the pretreatment phase to facilitate selective leaching, streamline downstream purification, and enable closed-loop hydrometallurgical processes.

Table 2 provides a critical comparison of the main pretreatment strategies reported from 2020 to 2025 for arsenic and bismuth control in Sb-bearing feedstocks, highlighting their underlying mechanisms, applicable feedstock types, and their direct effects on leaching selectivity and overall flowsheet complexity.

Table 2. Pretreatment strategies for arsenic and bismuth control in antimony hydrometallurgical processing (2020–2025).Adapted from Ling et al. (2022), Díaz et al. (2023), Benabdallah et al. (2023), Luo et al. (2024), and Alguacil (2025).

Pretreatment strategy	Primary mechanism	Target impurity	Typical feedstock	Impact on leaching selectivity	Key limitations / risks
Controlled oxidation (thermal or chemical)	Oxidation of As(III)/Sb(III) to less soluble forms	As, partially Bi	Stibnite ores, crude Sb_2O_3	Improves Sb/As separation window in alkaline or acidic systems	Risk of Sb encapsulation; formation of refractory oxides
Selective hydrolysis (pH-controlled)	Differential hydrolysis of Sb vs. As species	As	Copper electrorefining electrolytes, chloride leachates	High selectivity for Sb recovery when redox and pH are controlled	Narrow operating window; sensitive to solution chemistry
Selective precipitation	Formation of insoluble As/Bi compounds	As, Bi	Secondary residues, crude Sb oxides	Effective impurity removal prior to Sb recovery	Generates secondary residues; reagent consumption
Solvent extraction (integrated pretreatment)	Preferential complexation of As/Bi	Bi, partially As	Cu–Sb residues, hydrochloric solutions	Enhances downstream Sb purity	Limited selectivity for Sb; organic losses
Alkaline sulfide conditioning	Preferential dissolution of Sb over As	As	Stibnite tailings, complex sulfide ores	Improves Sb selectivity during leaching	Co-dissolution of As under aggressive conditions
Redox adjustment (Ehcontrol)	Stabilization of As(V) or Bi(III)	As, Bi	Mixed Sb-bearing leachates	Controls impurity mobility during leaching	Requires tight process control

As shown in Table 2, arsenic and bismuth control is most effective when implemented as an integrated pretreatment step rather than deferred to downstream purification alone. Across recent studies, strategies such as controlled oxidation, selective hydrolysis, and redox adjustment consistently emerge as defining operations, conditioning both Sb speciation and impurity behavior prior to leaching. At the same time, the table highlights a recurring limitation: gains in pretreatment selectivity are often accompanied by narrower operating windows, increased process complexity, and tighter control requirements. These trade-offs reinforce a central conclusion of this review—that successful antimony hydrometallurgical flowsheets depend on a deliberate balance among pretreatment intensity, leaching chemistry, and purification strategy, rather than on the optimization of any single unit operation in isolation.

4.3. Mechanical Activation and Granulometric Control

Mechanical activation and particle-size reduction have re-emerged as enabling pretreatment strategies for Sb-bearing materials, particularly in systems where mineralogical barriers impose diffusion-controlled leaching kinetics. Recent studies on milling, mechanical activation, and ultrasound-assisted processing consistently report accelerated Sb dissolution rates, lower apparent activation energies, and improved short-term extraction performance (Moosavi Nejad, 2020; Ran et al., 2024; Şayan & Çalışkan, 2024).

However, a critical review of the literature indicates that mechanical activation alone cannot overcome unfavorable mineralogical associations. In complex secondary materials—such as smelter slags, dusts, and glassy residues—increased surface area often raises reagent consumption without proportional gains in Sb recovery, unless coupled with chemical or thermal pretreatment that modifies phase accessibility (Ling et al., 2021; Yu et al., 2023). In these systems, the dominant limitation remains encapsulation within silicate or iron-rich matrices rather than intrinsic reaction kinetics.

Although mechanical activation and particle-size control are often presented as straightforward ways to enhance antimony leaching, recent evidence shows that their effectiveness is governed by mineralogical accessibility rather than surface area alone. While size reduction can shorten diffusion path lengths and increase apparent leaching rates, it does not inherently overcome encapsulation or phase shielding. Figure 6 conceptually illustrates how mechanical activation interacts with diffusion barriers, emphasizing that particle-size reduction must be evaluated alongside mineralogical conditioning and overall pretreatment intensity, rather than as an isolated optimization parameter.

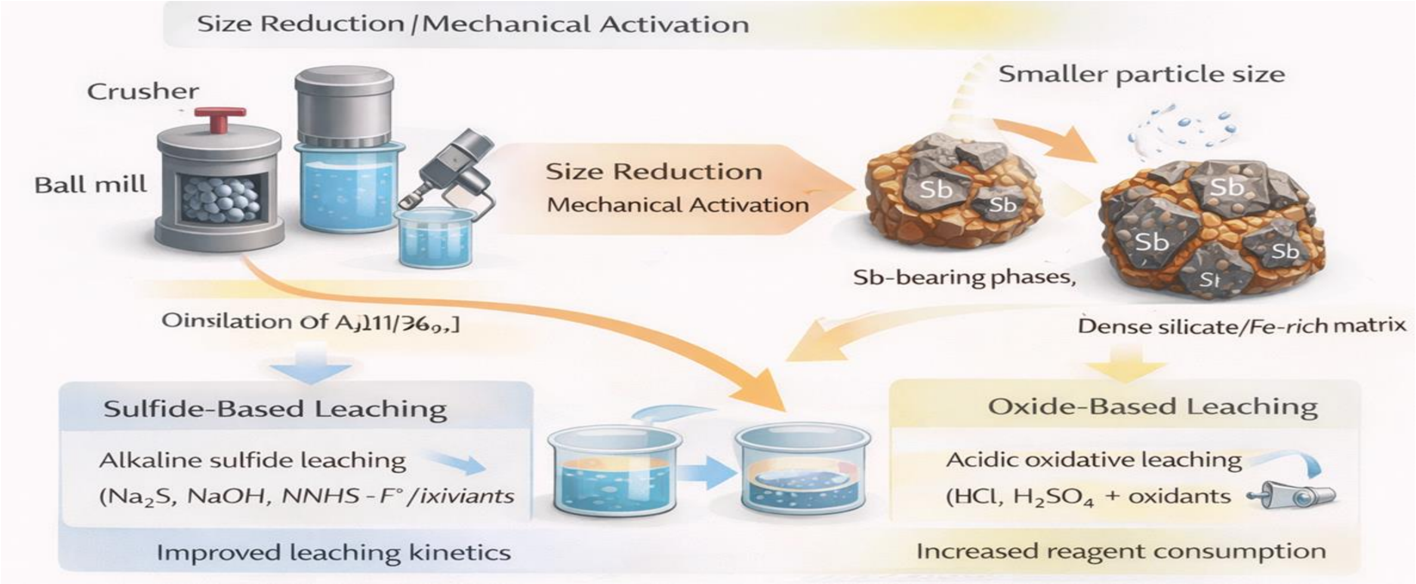

Figure 6. Effect of mechanical activation and granulometric control on antimony leaching kinetics and diffusion limitations. Adapted from Moosavi Nejad (2020), Ran et al. (2024), and Şayan & Çalışkan (2024).

Figure 6 shows that mechanical activation primarily affects kinetic accessibility rather than thermodynamic selectivity. Its effectiveness is therefore strongly dependent on prior mineralogical conditioning and must be evaluated within an integrated pretreatment–leaching–purification sequence rather than as a standalone enhancement strategy.

A recurring theme in the 2020–2025 literature is that many hydrometallurgical studies implicitly treat leaching chemistry as the dominant innovation, while the true determinants of process viability lie upstream, in pretreatment and impurity control. Management of oxidation state, selective stabilization or removal of arsenic and bismuth, and mineralogical conditioning consistently emerge as the steps that ultimately determine whether a given leaching system can operate selectively, sustainably, and at industrially relevant scales.

This observation reinforces the central argument developed in Section 3: hydrometallurgical antimony recovery must be evaluated as an integrated sequence—mineralogy → pretreatment → leaching → purification—rather than as isolated leaching reactions optimized in laboratory settings.

Pretreatment strategies therefore play a decisive role not by directly enhancing antimony recovery, but by defining the mineralogical accessibility and chemical speciation that ultimately govern leaching behavior. The effectiveness of any subsequent leaching system must thus be interpreted in light of the transformations induced during pretreatment, rather than evaluated in isolation.

Although pretreatment strategies are frequently presented as decisive breakthroughs, their true contribution lies in conditioning mineralogical accessibility rather than directly enhancing antimony recovery. Without clear linkage to downstream leaching behavior and impurity control, pretreatment performance metrics alone remain insufficient to support process-scale decision making.

5. Leaching Systems: Chemical Pathways, Selectivity, and Process Maturity

Leaching constitutes the first aqueous step in the hydrometallurgical flowsheet, translating pretreatment-induced mineralogical changes into chemically complex pregnant leach solutions (PLS). Accordingly, leaching performance must be assessed not only by extraction efficiency but also by the composition and stability of the resulting solutions. Building on the mineralogical constraints discussed in Section 3 and the pretreatment logic established in Section 4, leaching systems constitute the chemical core of antimony hydrometallurgy. Nevertheless, a critical assessment of the literature published between 2020 and 2025 indicates that leaching chemistry alone is rarely the decisive factor in process viability. In practice, extraction efficiency must be evaluated alongside selectivity for antimony over coexisting impurities, effluent composition, purification requirements, and operational risk, which collectively define the effective maturity of a given processing route.

This section provides a critical comparison of the main leaching systems reported for antimony recovery, organized by lixiviant chemistry and evaluated against dissolution mechanisms, selectivity, kinetic and mass-transfer limitations, operational and safety considerations, environmental footprint, and perceived process maturity, herein referred to as the technology readiness feel (TRF). Rather than focusing exclusively on reported extraction yields, the discussion emphasizes how well each leaching system integrates—or fails to integrate—downstream separation, effluent management, and impurity control.

To support a systematic and comparative assessment, Table 3 consolidates the dominant hydrometallurgical leaching systems for antimony reported from 2020 to 2025. The table contrasts extraction performance with selectivity, dominant reaction pathways, effluent complexity, and indicative TRF. This integrated perspective reveals recurring trade-offs between chemical aggressiveness and downstream processing burden, underscoring a central observation of this review: many leaching systems show high laboratory-scale efficiency, yet their practical applicability is ultimately constrained by purification complexity and environmental management requirements.

Table 3. Overview of hydrometallurgical leaching systems for antimony recovery. Adapted from: Dembele et al. (2022, 2025); Zekavat et al. (2021); Ling et al. (2021, 2024); Benabdallah et al. (2023); Alguacil (2025); Panayotova and Panayotov (2025).

Leaching system	Typical feedstock	Dominant dissolution mechanism	Sb extraction level	Selectivity vs. impurities (As, Fe, Pb, Bi)	Main operational risks	Effluent / downstream impact
Alkaline sulfide (Na_2S–NaOH)	Stibnite-rich ores, Sb flotation tailings, sulfide residues	Formation of soluble thioantimonate complexes	High	High (for Sb_2S_3)	Sulfide consumption, oxygen ingress, sulfur passivation	Sulfide- and As-bearing liquors; requires controlled oxidation/precipitation
Alkaline caustic (NaOH)	Oxidized Sb ores (Sb_2O_3/Sb_2O_4), selected secondary oxides	Hydroxyl-complex dissolution of Sb oxides	Moderate–High	Moderate	Limited applicability window; co-dissolution of Pb/As	Caustic effluents; hydrolysis control required
Acid oxidative (HCl/H_2SO_4 + oxidants)	Oxidized ores, anode slimes, smelter residues	Oxidative dissolution of Sb (III/V)	High	Low	Extensive impurity dissolution; redox control	High purification demand (hydrolysis, SX, adsorption)
Chloride-based / complexing systems	Complex secondary resources, Cu refining residues	Chloride complex formation, redox-dependent speciation	High	Low–Moderate	Corrosion; hydrolysis instability	Chloride-rich liquors; corrosion-resistant materials needed
Integrated or hybrid routes	Slags, dusts, mixed industrial residues	Coupled pretreatment tailored leaching	Variable	Variable (feed-dependent)	Process complexity; feed variability	Often optimized for closed-loop operation

As shown in Table 3, no single leaching system simultaneously maximizes extraction efficiency, selectivity, and operational simplicity across the full range of antimony-bearing feedstocks. Alkaline sulfide leaching is the most selective route for sulfide-dominated materials; however, its practical deployment is constrained by diffusion-controlled kinetics, sulfide consumption, and operational handling risks. By contrast, acid oxidative and chloride-based systems offer greater chemical robustness and broader applicability, yet they consistently shift the main process bottleneck from dissolution to purification and effluent treatment. The growing emphasis on integrated and hybrid processing routes in recent literature reflects a consensus that effective antimony recovery cannot rely on leaching optimization alone but requires the deliberate coupling of pretreatment, leaching chemistry, and downstream separation strategies.

5.1. Alkaline Sulfide Leaching (Na_2S–NaOH): The Most Selective Route for Sb_2S_3

Alkaline sulfide leaching remains the most extensively investigated and industrially relevant hydrometallurgical route for processing stibnite (Sb_2S_3), primarily because of its high chemical selectivity for antimony. In this system, Sb dissolves via the formation of soluble thioantimonate complexes, while a large fraction of gangue minerals and base metal sulfides remains essentially inert, enabling selective extraction under moderately alkaline conditions (Rusalev et al., 2020; Dembele et al., 2022; Dembele et al., 2025).

Recent studies show that this chemistry is not limited to primary stibnite ores but can be successfully applied to flotation tailings, smelter residues, and mixed sulfide feedstocks, reinforcing its status as a benchmark route for selective Sb recovery from sulfide-dominated materials (AKCIL et al., 2024; Luo et al., 2024). Consequently, alkaline sulfide leaching is often used as the reference system for evaluating alternative hydrometallurgical routes.

Despite its favorable selectivity, alkaline sulfide leaching has intrinsic limitations that constrain operational robustness. Kinetic studies consistently report diffusion-controlled behavior, commonly attributed to sulfur-rich passivation layers, secondary surface products, and/or incomplete liberation of Sb-bearing phases (Moosavi Nejad, 2020; Rusalev et al., 2020). In addition, sulfide consumption is highly sensitive to oxygen ingress, which promotes thiosulfate formation, reagent losses, and increased operational and safety risks. Effluent management further complicates process integration, particularly under aggressive leaching conditions where residual sulfide and partial arsenic co-dissolution may occur (Zhang et al., 2023).

Overall, although alkaline sulfide leaching of stibnite is widely recognized as the most selective hydrometallurgical route for antimony recovery, its apparent chemical simplicity masks a complex interplay between reaction chemistry and mass transport. Although thioantimonate formation provides a strong thermodynamic driving force for Sb dissolution, experimental evidence indicates that overall leaching performance is governed primarily by diffusion and surface passivation rather than intrinsic reaction kinetics. Figure 7 schematically illustrates the thioantimonate reaction pathway and highlights the diffusion-limited nature of alkaline sulfide leaching, clarifying why process optimization often requires careful control of particle size, oxygen exposure, and hydrodynamic conditions rather than further intensification of leaching chemistry.

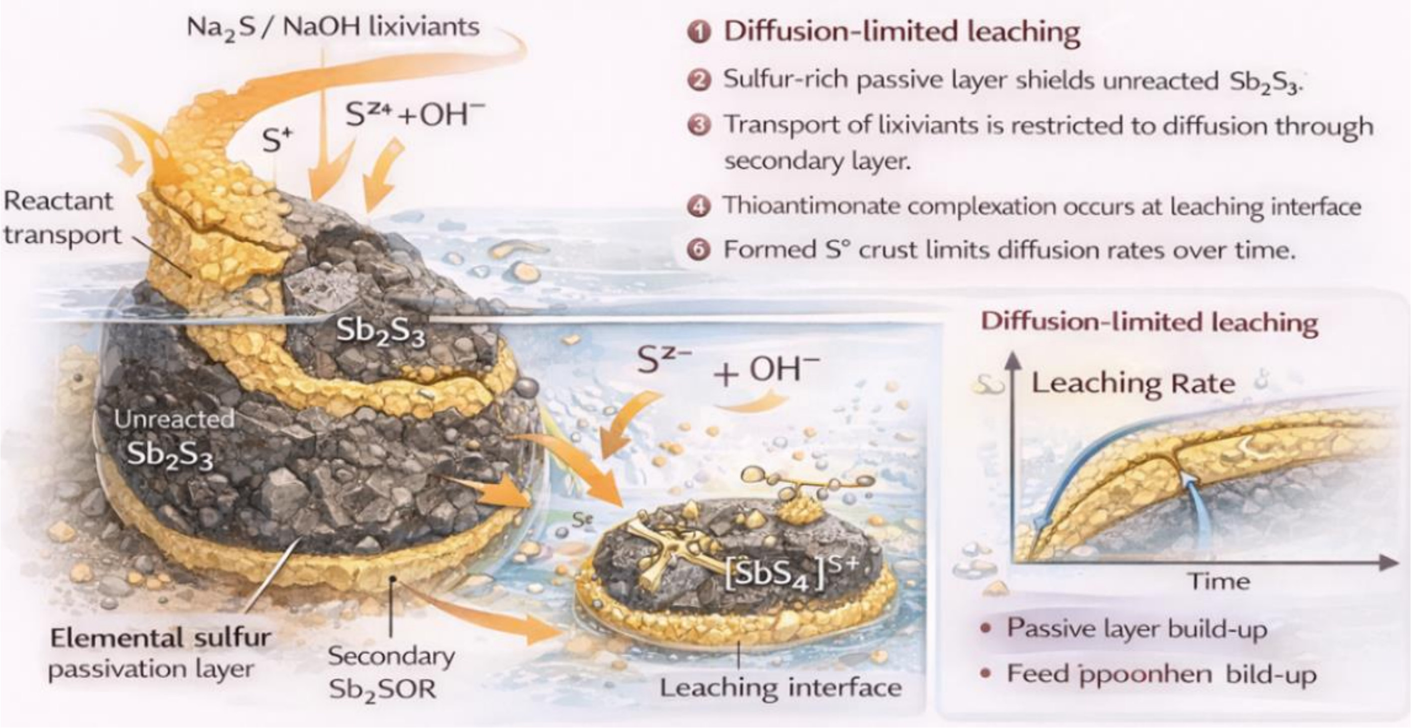

Figure 7. Reaction pathway for thioantimonate formation and diffusion-limited leaching behavior during alkaline sulfide processing of stibnite (Sb_2S_3).Adapted from Zekavat et al. (2021); Moosavi Nejad (2020); Dembele et al. (2022); Rusalev et al. (2020).

As illustrated in Figure 7, alkaline sulfide leaching proceeds by converting Sb_2S_3 into soluble thioantimonate species through sulfide complexation under strongly alkaline conditions. Despite favorable dissolution thermodynamics, the progressive accumulation of elemental sulfur and secondary surface layers restricts reagent access to the unreacted core, ultimately shifting the process toward diffusion-controlled kinetics. This behavior explains why increases in temperature, sulfide concentration, or agitation often yield diminishing returns beyond a critical threshold. Consequently, the practical performance of alkaline sulfide leaching is governed less by chemical equilibrium than by mass-transfer limitations and surface chemistry management. These constraints underscore the need to integrate pretreatment, redox control, and sulfide management when assessing the viability of this route beyond laboratory scale.

Technology readiness feel (TRF): Medium–High, reflecting strong chemical selectivity and demonstrated applicability, yet persistent challenges in reagent handling, oxygen sensitivity, and effluent control.

5.2. Alkaline Caustic Leaching (NaOH): Applicable to Oxide-Dominated Feeds

Alkaline caustic leaching with NaOH, without sulfide additives, has been extensively studied for oxidized antimony ores and oxide-rich secondary materials, where antimony occurs primarily as Sb_2O_3 or Sb_2O_4 (Zekavat et al., 2021; Zhang et al., 2023). In these systems, Sb dissolution proceeds through hydroxyl complex formation, thereby avoiding sulfide-related operational hazards and simplifying reagent handling.

Despite these advantages, the applicability of caustic leaching is intrinsically limited by feedstock mineralogy. While effective for relatively clean oxide materials, this route rapidly loses selectivity in the presence of iron oxides, silicate gangue, or complex polymetallic matrices, where antimony is commonly encapsulated or structurally bound within refractory phases (Ling et al., 2021; Ling et al., 2024). Under such conditions, caustic leaching often requires elevated temperatures or reagent concentrations, which promote the co-dissolution of lead and arsenic and shift the dominant process constraint from leaching to downstream purification.

Although alkaline sulfide and caustic leaching are often grouped under the broad “alkaline routes” for antimony recovery, their underlying mechanisms, selectivity windows, and operational risks differ fundamentally. Alkaline sulfide leaching relies on sulfur-based complexation and shows strong selectivity for Sb_2S_3, whereas caustic leaching is limited to oxide-rich feedstocks and remains highly sensitive to matrix effects. Table 4 provides a critical comparison of these two systems, highlighting how mineralogical context dictates their practical applicability and explaining why alkaline sulfide leaching continues to dominate for sulfide ores, while caustic leaching remains a niche solution for specific oxidized materials.

Table 4. Comparison between alkaline sulfide and alkaline caustic leaching systems for antimony recovery. Adapted from: Zekavat et al. (2021); Moosavi Nejad (2020); Dembele et al. (2022, 2025); Ling et al. (2021, 2024); Panayotova and Panayotov (2025).

Aspect	Alkaline sulfide leaching (Na_2S–NaOH)	Alkaline caustic leaching (NaOH)
Primary target phases	Sb_2S_3 (stibnite), sulfide-rich residues	Sb_2O_3, Sb_2O_4, selected oxidized residues
Dominant dissolution mechanism	Formation of soluble thioantimonate complexes	Hydroxyl complexation of Sb oxides
Selectivity toward Sb	High for sulfide-bound Sb	Moderate; highly feed-dependent
Selectivity vs. As	Limited (Sb–As co-dissolution common)	Often poor; As hydrolysis overlap
Kinetic control	Diffusion-limited due to sulfur passivation layers	Surface reaction and diffusion mixed control
Main operational risks	Sulfide consumption, oxygen ingress, H_2S evolution, sulfur passivation	Narrow applicability window; high caustic consumption
Sensitivity to mineralogy	High (requires sulfide-dominated feed)	Very high (requires oxide-dominated feed)
Downstream purification burden	Moderate; As and S management required	Moderate–high; hydrolysis control critical
Typical application scope	Primary ores, flotation tailings, sulfide residues	Oxidized ores, crude Sb_2O_3, selected secondary oxides
Technology readiness feel (TRF)	Medium–High	Medium

As summarized in Table 4, alkaline sulfide leaching offers superior selectivity for stibnite and sulfide-rich feedstocks but is intrinsically constrained by diffusion-limited kinetics and sulfur management challenges. By contrast, alkaline caustic leaching eliminates sulfide-related operational risks but has a markedly narrower applicability window, proving effective only when antimony is predominantly present as oxides and when gangue chemistry permits controlled hydrolysis. This comparison reinforces that neither alkaline route is universally applicable; rather, successful implementation depends on tight alignment among feedstock mineralogy, pretreatment strategy, and downstream purification requirements.

High antimony extraction yields are routinely reported across diverse leaching systems; however, these metrics often mask the simultaneous generation of chemically complex solutions enriched in problematic impurities. As a result, leaching efficiency, when evaluated in isolation, provides limited insight into the overall hydrometallurgical flowsheet's feasibility.

Technology readiness feel (TRF): Medium, reflecting chemical simplicity and operational familiarity, yet limited versatility across heterogeneous antimony-bearing materials.

5.3. Acid Oxidative Leaching (HCl/H_2SO_4 + Oxidants): Powerful but Purification-Driven

Acidic oxidative leaching systems, typically based on HCl or H_2SO_4 and combined with oxidizing agents such as Fe^{3+}, O_2, or H_2O_2, exhibit strong dissolution capacity and have been widely applied to oxidized antimony ores, anode slimes, and complex metallurgical residues (Díaz et al., 2023; Ibrahim et al., 2025). These systems are particularly attractive for feedstocks in which antimony occurs as oxides or in amorphous forms, and where alkaline routes are ineffective due to mineralogical constraints.

Despite their high extraction efficiency, acidic oxidative systems are inherently low in selectivity. Under strongly acidic and oxidizing conditions, antimony dissolution is invariably accompanied by extensive co-dissolution of arsenic, iron, lead, and bismuth, turning leaching into a preparatory step rather than a selective recovery operation (Benabdallah et al., 2023; Ling et al., 2024). As a result, overall process performance is often determined not by leaching yields but by the complexity and robustness of downstream purification strategies, including controlled hydrolysis, selective precipitation, solvent extraction, and solution recycling.

This intrinsic trade-off between dissolution efficiency and selectivity is a central limitation of acid oxidative leaching for antimony recovery. While aggressive chemical conditions enable rapid, extensive Sb solubilization, they also amplify impurity mobilization, effluent treatment requirements, and reagent consumption. Figure 8 illustrates this balance between antimony extraction and impurity co-dissolution, showing why purification and solution management—rather than leaching chemistry itself—often dominate capital and operating costs in acid-based processing routes.

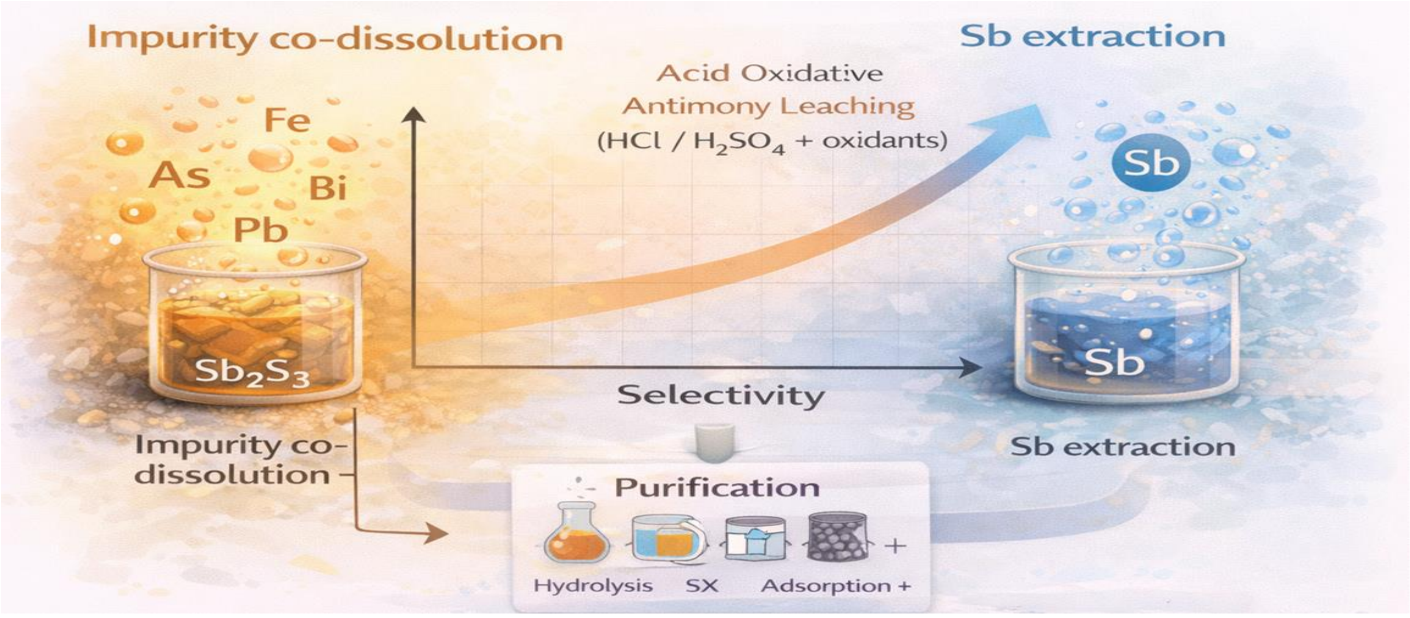

Figure 8. Trade-off between extraction efficiency and impurity co-dissolution in acid oxidative antimony leaching. Adapted from Díaz et al. (2023), Benabdallah et al. (2023), and Ling et al. (2024).

As illustrated in Figure 8, acid oxidative leaching routes inherently involve a selectivity–efficiency trade-off. Although high antimony extraction can be achieved under strongly acidic, oxidizing conditions, the concurrent dissolution of arsenic, iron, lead, and bismuth markedly increases solution complexity. Consequently, purification steps—rather than leaching itself—become the dominant contributors to reagent consumption, waste generation, and overall process cost. This recurring observation across studies published between 2020 and 2025 explains why acid oxidative systems, despite their apparent chemical robustness, often have limited industrial attractiveness unless coupled with highly selective, tightly controlled, and economically viable purification strategies.

Technology readiness feel (TRF): Medium, reflecting strong dissolution capability but heavy reliance on purification-intensive downstream processing.

5.4. Chloride-Based and Complexing Systems

Chloride-based leaching systems and complexing media have attracted growing attention for treating chemically complex secondary antimony resources, including copper electrorefining residues, anode slimes, and high-impurity Sb oxides (Cui et al., 2025; Luo et al., 2024). These systems leverage the strong complexation capacity of chloride ions to solubilize antimony under conditions where alkaline or sulfate-based routes are limited in effectiveness.

Despite their chemical versatility, chloride-based systems pose a distinct set of operational and environmental challenges. High chloride activity significantly increases corrosion risks, requires corrosion-resistant materials, and complicates equipment selection. In addition, antimony speciation in chloride media is highly sensitive to redox potential, chloride concentration, and pH, making hydrolysis control and selective precipitation particularly demanding (Díaz-Gutiérrez et al., 2025). Poorly controlled conditions can rapidly lead to Sb losses through premature hydrolysis or excessive co-dissolution of impurities, further increasing solution complexity.

From a process integration perspective, managing chloride effluent is a critical constraint, especially in jurisdictions with stringent discharge regulations. The need for closed-loop operation, chloride recovery, or neutralization substantially increases flowsheet complexity and capital requirements. Consequently, although chloride-based systems offer high chemical power and flexibility, their practical deployment remains limited to niche applications where feedstock complexity justifies the added operational burden.

Technology readiness feel (TRF): Low–Medium, reflecting strong dissolution capability and adaptability to complex residues, but high operational complexity, corrosion risk, and demanding effluent management.

5.5. Secondary Resources Focus: Leaching in a Circular Economy Context

Between 2020 and 2025, a marked increase in studies explicitly targeting secondary antimony-bearing resources—such as slags, dusts, spent electrolytes, and industrial by-products—has been observed (Ling et al., 2021; Yu et al., 2023; Panayotova & Panayotov, 2025). This shift reflects a broader conceptual transition in antimony hydrometallurgical research, moving away from a primary focus on sulfide ores toward the valorization of residues generated along mining, smelting, and refining value chains. The drivers of this transition extend beyond resource depletion concerns to include stricter environmental regulations, escalating impurity management challenges, and the strategic role of antimony within circular economy frameworks.

Across these studies, a consistent conclusion emerges: leaching strategies for secondary feedstocks cannot be generalized and must instead be residue-specific, explicitly integrated with pretreatment and downstream purification to enable closed-loop operation. In contrast to primary ores, secondary materials exhibit heterogeneous chemical signatures and complex phase associations, making leaching chemistry alone insufficient as a performance predictor. Figure 9 schematically captures the temporal and conceptual evolution of Sb leaching research between 2020 and 2025, highlighting the growing emphasis on system integration, impurity control, and circularity as defining features of contemporary antimony hydrometallurgy.

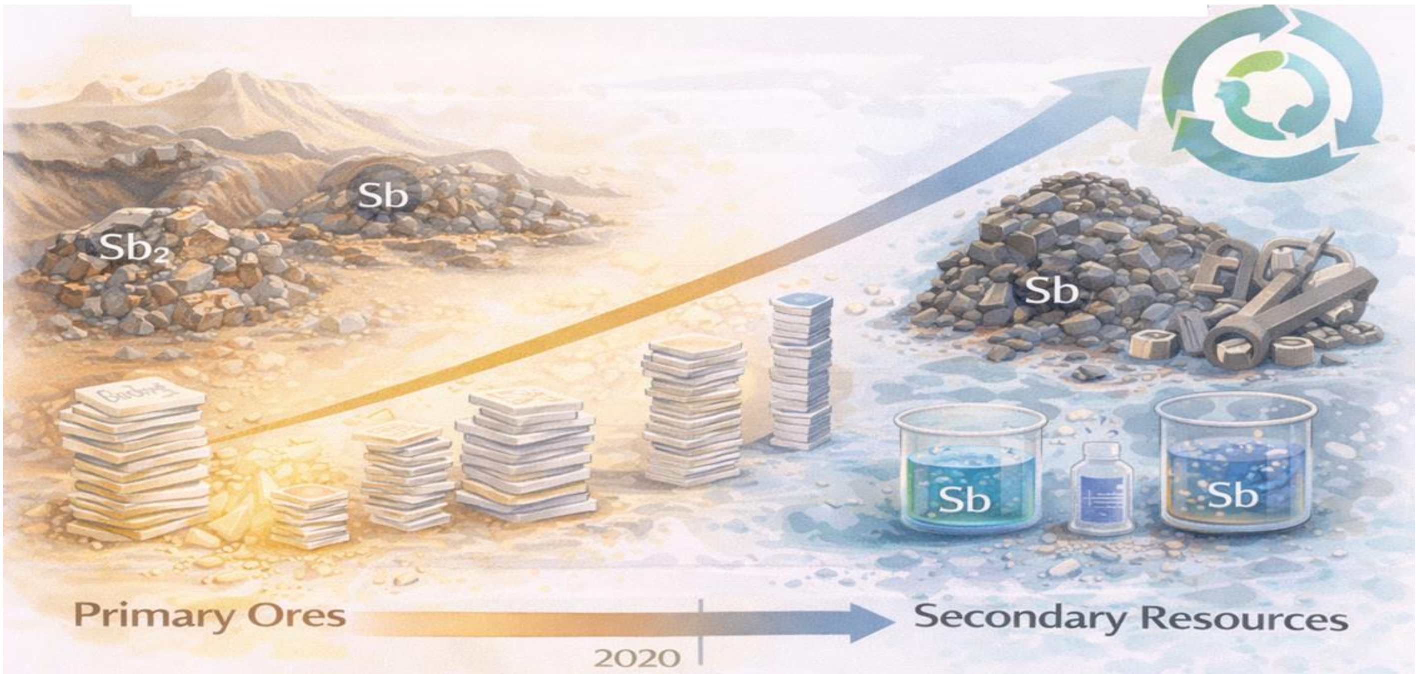

Figure 9. Conceptual evolution of antimony (Sb) leaching research focus from primary ores toward secondary resources within a circular economy framework. Adapted from: Ling et al. (2021); Yu et al. (2023); Alguacil (2025); Panayotova & Panayotov (2025).

As illustrated in Figure 9, recent literature shows a pronounced reorientation of antimony hydrometallurgical research priorities. Whereas earlier studies focused on stibnite-rich primary ores, emphasizing dissolution efficiency, more recent contributions increasingly focus on secondary resources with heterogeneous chemical signatures and complex mineralogical matrices. This shift reflects a growing recognition that secondary feedstocks—such as smelter slags, dusts, anode slimes, and spent electrolytes—present both a challenge and an opportunity: although antimony is often present at lower concentrations and in less accessible forms, these materials enable integrated Sb recovery coupled with impurity mitigation and waste minimization.

Importantly, Figure 9 shows that the principal scientific and technological bottleneck has progressively shifted from leaching chemistry alone to pretreatment design, impurity separation, and solution management. In studies of secondary resources, successful Sb recovery is often governed by the ability to control the behavior of arsenic, lead, and bismuth, stabilize effluents, and integrate purification steps into closed-loop flowsheets. As a result, contemporary Sb hydrometallurgical research increasingly evaluates process performance in terms of selectivity, environmental compatibility, and system integration rather than extraction yield in isolation.

Overall, Figure 9 reinforces a central conclusion of this review: future advances in antimony hydrometallurgy are likely to be driven less by incremental improvements in leaching reagents and more by holistic process concepts that align feedstock complexity, pretreatment strategies, and purification logic within circular-economy–oriented frameworks.

Technology readiness feel (TRF): Emerging, reflecting high sustainability relevance and strategic importance, yet with strong dependence on feedstock heterogeneity and system integration.

Across the leaching systems reviewed, a consistent conclusion emerges: antimony dissolution is rarely the limiting step, whereas impurity co-dissolution and solution complexity dominate downstream challenges. Consequently, the viability of leaching strategies can only be judged in conjunction with their implications for purification, separation efficiency, and final product quality.

The literature consistently indicates that antimony hydrometallurgy is seldom constrained by dissolution chemistry, whereas downstream selectivity and impurity management dominate both technical risk and cost structure. Consequently, leaching strategies should be regarded as preparatory steps whose success is ultimately validated—or invalidated—during purification.

6. Purification and Separation from Pregnant Leach Solutions (PLS): Where Most Flowsheets Fail

Purification is not a downstream auxiliary operation but the central determinant of antimony hydrometallurgical viability, governing product quality, environmental compliance, and technology readiness.

Purification and separation from pregnant leach solutions represent the most critical and failure-prone stages in antimony hydrometallurgical flowsheets, as they determine selectivity, impurity control, and the feasibility of downstream recovery routes.

Across the 2020–2025 literature, purification challenges are consistently linked to three recurring issues: (i) overlapping chemical behavior of Sb with As and Bi, (ii) sensitivity of Sb speciation to redox and pH conditions, and (iii) generation of secondary effluents that compromise closed-loop operation (Benabdallah et al., 2023; Ling et al., 2024; Panayotova & Panayotov, 2025). As a result, purification must be evaluated not as an auxiliary step but as a core unit operation that ultimately determines whether Sb recovery routes are industrially viable.

To frame this discussion, Table 5 summarizes the primary purification and separation strategies applied to Sb-bearing PLS from 2020 to 2025, highlighting their mechanisms, selectivity, maturity, and common failure points.

Table 5. Comparison of purification and separation routes for Sb-bearing pregnant leach solutions (PLS) reported in the 2020–2025.Adapted from: Benabdallah et al. (2023); Díaz et al. (2023); Díaz-Gutiérrez et al. (2025); Hernández-Pérez et al. (2023); Luo et al. (2024); Ling et al. (2022, 2024); Panayotova & Panayotov (2025); Garrido et al. (2025); Sudová et al. (2024); Spooren (2025); Ibrahim et al. (2025).

Purification family	Typical unit operation(s)	Target Sb species (dominant)	Selectivity vs As	Selectivity vs Bi	Regeneration / reusability	Typical product form
Hydrolysis / selective precipitation	pH adjustment; staged hydrolysis; seed-assisted precipitation	Sb (III) in chloride/acid media; sometimes Sb (V) after redox tuning	Medium (overlap frequent)	Low–Medium (Bi co-precipitation common)	High (simple, but consumes reagents; not "regeneration")	Sb_2O_3 / Sb oxy-hydroxide solids
Direct chemical precipitation (non-hydrolytic)	Sulfide precipitation; selective precipitation by counter-ions	Sb (III)/Sb (V) depending on chemistry	Variable	Variable	Medium	Mixed Sb salts/precipitates
Solvent extraction (SX)	Extraction + scrubbing + stripping; redox conditioning	Sb (III) usually preferred; Sb (V) harder	Medium (system-dependent)	Low–Medium (Bi co-extraction frequent)	Medium–High (organic reusable, but degradation possible)	Concentrated Sb strip liquor; Sb_2O_3 after precipitation
Ion exchange (IX) / resins	Fixed-bed adsorption; elution/regeneration; polishing stage	Often Sb(V) anionic complexes; Sb(III) system-dependent	Medium–High (can be tuned)	Medium	High (regeneration feasible)	Eluate enriched in Sb; final Sb product via precipitation
Adsorption (engineered sorbents)	Sorption on oxides/carbons/hybrids; desorption trials	Sb(V) often stronger; Sb(III) variable	Medium	Medium	Low–Medium (regeneration often not demonstrated)	"Removed Sb" on sorbent; sometimes eluate
Electrochemical recovery (electrowinning / electrode position)	Electrodeposition; membrane electrolysis; electro-electrodialysis	Sb(III) typical; depends on electrolyte	Medium (depends on speciation)	Medium	High (electricity-driven; continuous possible)	Sb metal or Sb oxide (depending)
Membrane electrolysis / electro-electrodialysis	Membrane cells; selective transport + regeneration of media	Sb species depends on media	Medium	Medium	High (media recycling possible)	Regenerated leachant + Sb-enriched stream
Hybrid purification (staged: hydrolysis + SX/IX/EC)	Multi-step cascades	Mixed (Sb(III)/Sb(V))	High (best potential)	Medium–High		

As shown in Table 5, hydrolysis and precipitation are the most practical recovery routes due to operational simplicity and relative maturity, yet they remain highly sensitive to impurity co-mobilization and narrow pH/Eh selectivity windows (Díaz et al., 2023; Ibrahim et al., 2025). In contrast, SX and IX can deliver higher selectivity and greater concentration factors but introduce additional complexity through speciation control, corrosion, and multi-step circuit requirements (Benabdallah et al., 2023; Luo et al., 2024). Adsorption has rapidly expanded as a research topic, but most studies still demonstrate removal rather than true recovery, failing to demonstrate regeneration and the production of a saleable Sb product (Sudová et al., 2024; Spooren, 2025). Electrochemical routes and membrane-assisted variants are promising for closed-loop concepts, yet remain constrained by process-control demands and limited validation under real industrial PLS conditions (Hernández-Pérez et al., 2023; Garrido et al., 2025).

6.1. Hydrolysis and Selective Precipitation

Hydrolysis and selective precipitation remain the most widely used and industrially practical routes for antimony recovery from PLS, particularly for producing Sb_2O_3 and related salts. These methods are extensively reported for chloride-based and acidic leachates derived from copper electrorefining residues, anode slimes, and oxide-rich feedstocks (Díaz et al., 2023; Díaz-Gutiérrez et al., 2025; Panayotova & Panayotov, 2025).

Controlled pH adjustment enables Sb precipitation via hydrolysis, while arsenic and bismuth may remain partially soluble depending on redox conditions and the complexation environment (Benabdallah et al., 2023). This approach is attractive for its simplicity, low capital intensity, and compatibility with existing industrial circuits. However, its selectivity window is narrow, and minor deviations in pH or Eh often lead to co-precipitation, loss of product purity, or unstable solids (Ling et al., 2022; Ibrahim et al., 2025).

A critical limitation repeatedly observed in the literature is that hydrolysis-based recovery often yields mixed or poorly defined precipitates that require additional purification or thermal treatment to meet commercial specifications. Consequently, although hydrolysis is often presented as a "final recovery step," it often serves in practice as an intermediate concentration stage rather than a complete separation solution.

TRF assessment: High (simple, industrially proven, but impurity-sensitive).

6.2. Solvent Extraction (SX)

Solvent extraction has been extensively studied as a selective purification method for antimony-bearing pregnant leach solutions, particularly in chloride-rich media where Sb speciation can be tuned between Sb(III) and Sb(V). A variety of extractant families—including organophosphorus compounds, amines, and solvating reagents—have been evaluated for separating antimony from Cu-, Bi-, and As-bearing solutions (Benabdallah et al., 2023; Luo et al., 2024).

Recent studies consistently show that the oxidation state of antimony exerts first-order control over extraction behavior. Sb(III) generally exhibits higher extractability than Sb(V), enabling selective separation under carefully controlled redox and chloride conditions (Díaz-Gutiérrez et al., 2025). However, this apparent selectivity is often compromised by the co-extraction of bismuth and, in some systems, arsenic, which share overlapping coordination chemistry. Mitigation strategies typically involve multistage scrubbing, redox adjustment, or selective stripping, substantially increasing flowsheet complexity and reagent consumption (Cui et al., 2025).

Despite its strong theoretical basis, solvent extraction performance is often overestimated in laboratory-scale studies. When applied to real-world industrial PLS, SX systems frequently suffer from solvent degradation, poor phase disengagement, and corrosion-related constraints linked to high chloride concentrations. These factors limit operational robustness and long-term solvent stability, particularly under continuous operation.

As conceptually illustrated in Figure 10, effective Sb separation by SX requires tight control of oxidation state, chloride activity, and impurity speciation. Consequently, solvent extraction is best positioned as an intermediate purification step rather than a standalone recovery solution within integrated antimony hydrometallurgical flowsheets.

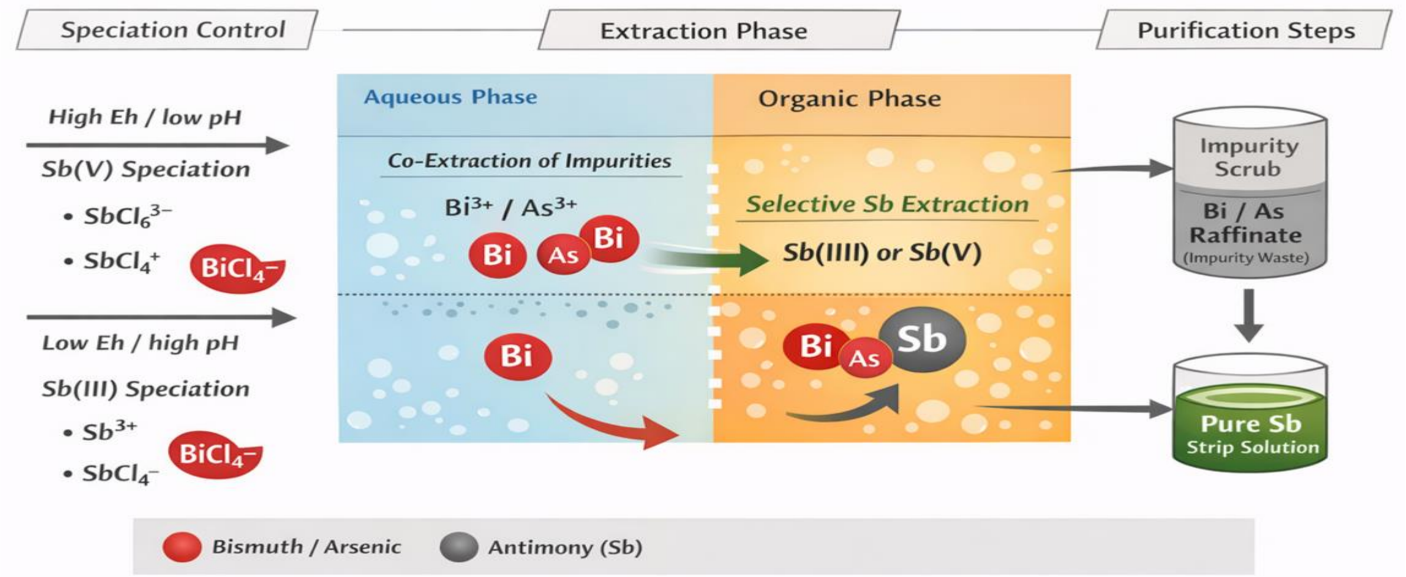

Figure 10. Conceptual representation of Sb speciation-dependent solvent extraction behavior and co-extraction challenges in chloride-based PLS. Adapted from: Benabdallah et al. (2023) and Luo et al. (2024).

As shown in Figure 10, solvent extraction performance is governed primarily by antimony speciation rather than extractant strength alone. Although both Sb(III) and Sb(V) can be efficiently transferred to the organic phase under chloride-rich conditions, arsenic and bismuth often co-extract unless redox potential, chloride activity, and pH are tightly controlled. This behavior explains why solvent extraction circuits for antimony often require multiple scrubbing and stripping steps, increasing operational complexity. Consequently, SX-based purification is best suited for applications where precise solution control is feasible and where high-purity Sb products justify the added process sophistication.

TRF assessment: Medium (high selectivity potential, operationally complex).

6.3. Ion Exchange (IX) and Resin-Based Systems

Ion exchange and functionalized resins have been increasingly explored for Sb recovery from industrial streams, particularly as polishing steps to meet regulatory or product-grade specifications. Applications include copper electrorefining electrolytes, dilute wash waters, and post-precipitation effluents (Hernández-Pérez et al., 2023; Garrido et al., 2025).

IX systems offer high selectivity under controlled conditions and can be regenerated multiple times, making them attractive for low-concentration streams. However, resin fouling, limited capacity in highly contaminated solutions, and sensitivity to competing anions limit their use as primary recovery steps (Martí-Calatayud et al., 2023).

Consequently, ion exchange is best understood as a finishing or polishing technology rather than a core separation method in Sb hydrometallurgy.

TRF assessment: Medium–High (industrially accepted, but capacity-limited).

6.4. Adsorption-Based Approaches

Between 2024 and 2025, adsorption has emerged as a rapidly expanding research focus for antimony separation from dilute, chemically complex aqueous streams, particularly in studies framed within environmental remediation and circular economy contexts (Sudová et al., 2024; Spooren, 2025; Xie et al., 2025). A broad range of adsorbent materials—including metal oxides, functionalized carbons, bio-based sorbents, and hybrid composites—has been proposed, often showing high Sb uptake capacities and apparent tolerance to competing ions.

However, a critical distinction must be drawn between antimony removal and antimony recovery. A substantial fraction of adsorption-based studies report high removal efficiencies without addressing adsorbent regeneration, Sb desorption, or conversion into a commercially viable product (Periferakis et al., 2022; Zhao et al., 2022). In such cases, adsorption serves as an environmental control measure rather than a resource recovery strategy.

This conceptual ambiguity significantly limits the applicability of adsorption as a primary recovery route. Without demonstrated regeneration cycles, eluate conditioning, and downstream Sb product formation, most adsorption systems remain confined to wastewater treatment, effluent polishing, or compliance-driven applications. As conceptually illustrated in Figure 11, adsorption-based processes often immobilize Sb from solution yet fail to close the material loop required for circular hydrometallurgical flowsheets.

Only adsorption strategies that explicitly integrate desorption, adsorbent reuse, and controlled Sb recovery pathways can be realistically considered beyond low-TRL environmental applications.

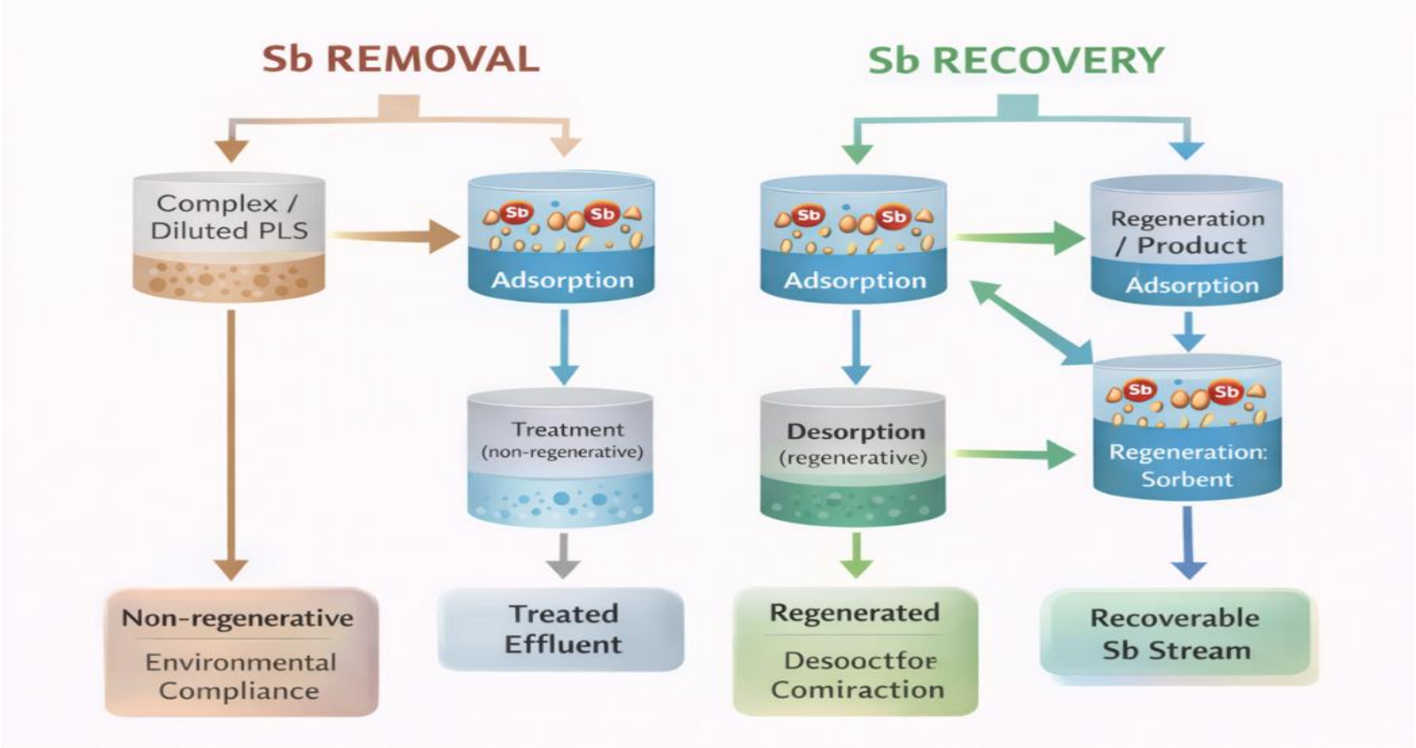

Figure 11. Distinction between antimony removal and true recovery in adsorption-based purification systems.Adapted from: Sudová et al. (2024) and Spooren (2025).

As illustrated in Figure 11, many adsorption-based studies published between 2020 and 2025 focus predominantly on antimony removal, emphasizing high uptake capacity and rapid kinetics but without demonstrating effective sorbent regeneration or recovery of a marketable Sb product. In such cases, adsorption functions primarily as a polishing or stabilization step rather than a true recovery operation. In contrast, regenerative adsorption pathways—where Sb can be selectively desorbed, concentrated, and recycled—offer a more sustainable route aligned with circular-economy principles, albeit at the cost of increased process complexity. This distinction highlights a recurring gap in the literature: while adsorption is increasingly accepted as a viable purification technology, its contribution to antimony valorization critically depends on regeneration efficiency, sorbent stability, and integration with downstream recovery steps.

TRF assessment: Emerging (strong academic progress but limited industrial validation).

Across all purification families, the literature converges on a critical insight: the technical success of antimony hydrometallurgical flowsheets is rarely constrained by leaching efficiency but rather by the ability to selectively recover Sb from complex PLS while managing impurities and effluents. Hydrolysis and precipitation dominate industrial practice for their simplicity, whereas solvent extraction, ion exchange, and adsorption offer higher selectivity at the cost of greater complexity and narrower operating windows.

Most importantly, many reported purification strategies implicitly focus on removal rather than recovery and fail to demonstrate regeneration, product specification, or closed-loop feasibility. These shortcomings underscore the need to evaluate purification strategies not in isolation but as integral components of fully coupled mineralogy–pretreatment–leaching–purification flowsheets.

These observations demonstrate that purification performance is governed not only by chemical selectivity but also by solution stability, reagent regeneration potential, and compatibility with final recovery routes. The implications of these constraints for product quality and technology readiness are examined in the following section

Despite extensive laboratory-scale investigations, many purification strategies remain weakly validated at the flowsheet level, with limited discussion of reagent regeneration, impurity recycling, and long-term operational stability. These gaps significantly constrain technology readiness and impede realistic scale-up assessments.

7. Final Recovery: Sb Products and Electrochemical Routes

Final recovery should not be treated as a downstream formality but as the decisive validation step of antimony hydrometallurgical flowsheets. Without clear demonstration of product quality, stability, and market relevance, even highly selective leaching and purification strategies remain academically attractive yet industrially unconvincing.

7.1. Target Products and Quality Requirements

Hydrometallurgical antimony processing typically targets three main product categories: (i) antimony trioxide (Sb_2O_3), (ii) metallic antimony, and (iii) antimony salts, including pyroantimonates and thioantimonate derivatives. Among these, Sb_2O_3 remains the most widely reported and industrially relevant product, with broad applications in flame retardants, catalysts, and specialty chemicals (Majzlan, 2021; Ling et al., 2022).

Sb_2O_3 is most commonly recovered by controlled hydrolysis or precipitation from purified Sb-bearing solutions, often after oxidative or chloride-based leaching. Although this route is often described as straightforward, recent studies show that product purity is highly sensitive to residual As, Bi, Pb, and Fe in solution (Díaz et al., 2023; Ling et al., 2024). Even trace contamination can render the product unsuitable for chemical-grade applications, underscoring the central role of upstream purification.

Metallic antimony recovery is less common but remains attractive for applications in alloys and electronics. Hydrometallurgical routes to Sb metal typically rely on electrowinning or chemical reduction from highly purified solutions (Rusalev et al., 2023; Luo et al., 2024). However, the narrow impurity tolerance of electrochemical systems significantly limits their applicability in complex or poorly conditioned PLS.

Antimony salts, including sodium pyroantimonate and crystalline thioantimonates, are often produced as intermediate or niche products, particularly in alkaline sulfide systems (Moosavi Nejad, 2020; Zhang et al., 2023). Although these compounds can be isolated with relatively high selectivity, their market scope is limited, and long-term stability and handling considerations are rarely addressed in experimental studies.

Although leaching and purification steps are widely reported in the antimony hydrometallurgical literature, far fewer studies provide a clear, systematic discussion of the final products obtained and their compliance with commercial specifications. To highlight this gap and enable cross-comparison, Table 6 summarizes the main antimony products reported from 2020 to 2025, linking product form, purity requirements, precursor PLS characteristics, and intended applications.

Table 6. Overview of final antimony products reported in hydrometallurgical studies Adapted from: Moosavi Nejad (2020); Rusalev et al. (2020, 2023); Ling et al. (2022, 2024); Díaz et al. (2023); Luo et al. (2024); Panayotova & Panayotov (2025); Cui et al. (2025).

Final Sb product	Typical recovery route	Precursor PLS requirements	Purity targets (reported)	Main impurities of concern	Typical applications
Sb_2O_3 (chemical grade)	Hydrolysis / selective precipitation	Sb(III)/Sb(V) solution, low As/Bi/Pb	>99.0–99.5 wt% Sb_2O_3	As, Bi, Pb, Fe	Flame retardants, catalysts, pigments
Metallic Sb	Electrowinning / chemical reduction	Highly purified PLS, controlled speciation	>99.5 wt% Sb (lab scale)	As, Bi, Cu	Alloys, electronics, battery materials
Sodium pyroantimonate	Alkaline precipitation / crystallization	Alkaline Sb(V)-rich solutions	Typically reagent-grade	Na, As	Specialty chemicals, analytical reagents
Thioantimonate salts	Crystallization from sulfide media	Alkaline sulfide leachates	Variable (often unspecified)	As, S	Intermediate products, niche uses
Sb-loaded adsorbents	Adsorption (non-regenerative)	Dilute or complex PLS	Not applicable (removal-based)	—	Effluent treatment
Concentrated Sb eluates	Regenerative adsorption / IX	Dilute PLS with selective binding	Solution concentrate	As, Bi	Feed for downstream recovery

As shown in Table 6, Sb_2O_3 remains the most consistently reported and industrially relevant product obtained via hydrometallurgical routes, largely because of its relatively simple recovery by hydrolysis and broad commercial applicability. However, even for this seemingly mature product, stringent control of arsenic and bismuth is repeatedly identified as the decisive factor in product acceptance.

In contrast, metallic antimony and electrochemically derived products have higher value but significantly narrower operating windows, requiring exceptionally clean precursor solutions. Many studies report promising laboratory-scale yields yet stop short of demonstrating long-term stability, impurity tolerance, or scalability. Finally, a substantial fraction of the literature focuses on Sb removal rather than recovery, particularly in adsorption-based systems, where the absence of regeneration or product isolation limits true resource valorization.

Overall, Table 6 reinforces a central conclusion of this review: successful hydrometallurgical antimony processing cannot be judged solely by leaching or purification efficiency but must ultimately be validated by the production of a specified, marketable Sb product.

7.2. Electrowinning and Electrochemical Recovery Routes

Alongside conventional hydrometallurgical recovery routes, a subset of recent studies has explored electrochemical, vacuum-based, and molten-salt approaches for antimony separation and purification. These routes are often presented as high-selectivity or high-purity alternatives, particularly for complex feeds and impurity-rich systems. However, a critical assessment of the literature indicates that most of these technologies remain confined to laboratory or conceptual scales, with limited demonstration of integration into complete hydrometallurgical flowsheets. Electrochemical routes reported between 2020 and 2025 span electrowinning, membrane electrolysis, and intensified electrolysis applied to spent electrolytes, low-grade leachates, and copper-refining process streams. Yet they consistently rely on highly conditioned solutions and tightly controlled operating windows, limiting their transferability to industrial pregnant leach solutions (Thanu & Jayakumar, 2020; Sajadi et al., 2024; Tian et al., 2025; Garrido et al., 2025).

Electrochemical separation strategies have been proposed for selective Sb recovery from polymetallic systems, including lead–antimony–arsenic alloys and secondary residues, with reported advantages in separation efficiency under tightly controlled conditions (Cao et al., 2025; Wei et al., 2025). Nevertheless, these approaches typically require highly conditioned feeds, strict redox control, and narrow compositional windows, significantly limiting their applicability to real pregnant leach solutions without extensive upstream purification.

Vacuum- and gas-phase separation routes have also been investigated as purification methods for crude antimony or Sb-rich intermediates, showing promising selectivity over certain metallic impurities (Meng et al., 2024a; Meng et al., 2024b; Zhou et al., 2021). Despite these advantages, such processes are inherently energy-intensive and commonly rely on pyrometallurgical preconditioning or high-temperature operation, placing them outside the core scope of low-temperature hydrometallurgical processing.

Molten salt electrolysis and related high-temperature electrochemical methods have attracted attention as potentially direct pathways for antimony extraction or desulfurization from concentrates and complex residues (Zhu et al., 2024; Xu et al., 2025). While these studies provide valuable mechanistic insights and demonstrate high extraction efficiencies under controlled conditions, their reliance on molten media, elevated temperatures, and specialized materials raises significant challenges related to scalability, materials compatibility, and environmental management.

Overall, electrochemical, vacuum-based, and molten-salt routes should be regarded as promising but niche technologies within the antimony processing landscape. At present, their relevance lies primarily in targeted purification or specialty applications rather than as broadly deployable alternatives to established hydrometallurgical recovery schemes. From a technology readiness perspective, these routes generally exhibit low-to-medium maturity and require substantial further development to demonstrate compatibility with upstream leaching and downstream product specification requirements.

7.3. Critical Assessment of Final Recovery Performance

Across all final recovery routes, a recurring issue in the literature is conflating Sb removal with Sb recovery. Numerous studies report high removal efficiencies from solution but stop short of demonstrating product isolation, reagent regeneration, or production of a material meeting commercial specifications (Panayotova & Panayotov, 2025; Ling et al., 2024). This distinction is particularly critical for adsorption- and precipitation-based systems, where Sb may be immobilized rather than valorized.

From a technology readiness perspective, Sb_2O_3 precipitation remains the most mature and industrially transferable route, provided upstream purification is adequate. Electrowinning and electrochemical methods show strong potential but currently fall into a lower TRF category because of their sensitivity to feed quality and operational complexity.

Overall, final recovery should not be treated as a downstream formality but as a decisive validation step for hydrometallurgical flowsheets. Without clear demonstration of product quality, stability, and market relevance, even highly selective leaching and purification strategies may remain academically attractive yet industrially unconvincing.

The analysis in Sections 6 and 7 shows that purification and final recovery—not leaching—constitute the dominant failure points in most antimony hydrometallurgical flowsheets reported from 2020 to 2025. The following section synthesizes these observations to identify system-level bottlenecks, research gaps, and future perspectives, with particular emphasis on bridging the gap between laboratory-scale selectivity and industrial-scale robustness.

8. Residues, Effluents, and EHS: The Hidden Bottlenecks of Sb Hydrometallurgy

While leaching, purification, and final product recovery dominate the technical narrative of antimony hydrometallurgy, residues and effluents often determine the environmental acceptability, regulatory compliance, and long-term viability of proposed flowsheets. A critical review of studies published between 2020 and 2025 reveals a recurring pattern: many hydrometallurgical routes report high Sb recovery efficiencies while simultaneously producing chemically unstable or environmentally problematic residues, effectively shifting the process burden from metal recovery to waste and effluent management. This “burden shifting” has been explicitly highlighted in substance flow analyses of antimony metallurgical systems, which show that incomplete integration of residue and effluent management merely relocates environmental risk across process stages rather than eliminates it (Ke, 2023).

This section critically examines the three most persistent EHS challenges identified in recent antimony hydrometallurgical studies: (i) sulfide-bearing alkaline effluents, (ii) chloride-rich process circuits, and (iii) arsenic stabilization and disposal. Across all cases, the literature converges on a central conclusion: the failure to explicitly integrate residue and effluent management into flowsheet design remains a primary reason that laboratory-scale successes in Sb hydrometallurgy rarely translate into robust industrial implementation. This limitation is further compounded by the frequent absence of system-level environmental assessment, as illustrated by life-cycle–based studies of related hydrometallurgical systems, which caution that EHS claims cannot be substantiated without clearly defined system boundaries and downstream fate analysis (Karal et al., 2021; Ke, 2023).

Concrete examples from recent antimony-processing studies illustrate how these challenges manifest in practice. Acidic effluents containing both antimony and arsenic, for instance, have been successfully treated by selective reduction and evaporative crystallization; however, these approaches also underscore the need for careful control of secondary residues and long-term stability of recovered solids (Tian et al., 2020). Similarly, high-arsenic antimony oxide powders and polymetallic residues highlight the complex co-behavior of Pb, As, and Sb during leaching and purification, where selective metal recovery is inseparable from impurity stabilization (Shen et al., 2024; Yang et al., 2025). The influence of lead on antimony dissolution and solution chemistry further shows that mixed-metal systems cannot be evaluated using single-element performance metrics alone, as impurity interactions directly affect both process efficiency and effluent composition (Hirata-Miyasaki & Anderson, 2025).

Together, these case anchors show that EHS considerations in antimony hydrometallurgy are not peripheral constraints but core determinants of process viability. Flowsheets that do not explicitly demonstrate stable residue formation, controlled effluent chemistry, and impurity management across Pb–As–Sb systems should therefore be considered environmentally incomplete, regardless of their reported extraction efficiencies (Karal et al., 2021).

8.1. Sulfide-Bearing Effluents in Alkaline Leaching Systems

Alkaline sulfide leaching (Na_2S–NaOH), while highly selective for stibnite, inevitably produces effluents containing residual sulfide, polysulfides, thiosulfate, and dissolved arsenic species. If inadequately managed, these streams pose significant environmental and operational risks, including acute toxicity, odor generation, corrosion, and chemical instability during discharge or internal recycling (Rusalev et al., 2020; Dembele et al., 2022; Dembele et al., 2025).

Recent studies consistently indicate that controlled oxidation of sulfide-bearing liquors is the most viable mitigation strategy, enabling the conversion of dissolved sulfide to elemental sulfur or sulfate before discharge or reuse (Moosavi Nejad, 2020; Zhang et al., 2023). However, the literature also highlights a critical operational trade-off: excessive oxidation increases sulfate loading, reagent consumption, and scaling potential, whereas insufficient oxidation leads to sulfide breakthrough, safety hazards, and regulatory non-compliance.

To address these challenges, several authors propose partial circuit closure strategies that recycle oxidized effluents to leaching or pretreatment stages, thereby reducing fresh reagent demand and minimizing waste generation (Rusalev et al., 2020; Panayotova & Panayotov, 2025). Despite their conceptual appeal, most published flowsheets stop short of providing quantitative sulfur mass balances, speciation control, or long-term effluent stability assessments, underscoring a persistent disconnect between laboratory-scale demonstrations and industrially realistic environmental performance.

Overall, sulfide management is often treated as a secondary operational consideration, yet it is a first-order constraint on the scalability and regulatory acceptability of alkaline sulfide leaching systems. Flowsheets that do not explicitly demonstrate robust sulfide stabilization pathways, controlled oxidation logic, and effluent fate should therefore be considered environmentally incomplete, regardless of their reported leaching selectivity or extraction efficiency.

8.2. Chloride Circuits: Corrosion, Purge Streams, and System Compatibility

Chloride-based leaching and purification systems have gained prominence in processing complex secondary resources, particularly copper electrorefining residues, anode slimes, and high-impurity Sb oxides (Cui et al., 2025; Luo et al., 2024; Díaz-Gutiérrez et al., 2025). Although chemically powerful, chloride circuits pose a distinct set of EHS challenges.

High chloride concentrations accelerate equipment corrosion, requiring specialized construction materials and increasing CAPEX (Benabdallah et al., 2023; Luo et al., 2024). Moreover, chloride accumulation necessitates periodic purge streams that concentrate metals and halides and require dedicated treatment before disposal.

Compatibility issues also arise when chloride-rich liquors are coupled with solvent extraction or ion exchange. Many extractants and resins exhibit reduced lifetime or selectivity in high-Cl^- environments, increasing operating costs and waste generation (Luo et al., 2024; Vinardell et al., 2024).

Chloride systems are often described as flexible and robust, yet their environmental footprint is highly sensitive to purge management and material selection. Without demonstrated chloride control strategies, these approaches risk shifting complexity from metallurgy to waste handling.

8.3. Arsenic Management: Stabilization vs. Problem Transfer

Arsenic management is the most critical and recurrent EHS challenge in antimony hydrometallurgy. Across both primary ores and secondary resources, Sb and As exhibit overlapping dissolution behavior, resulting in unavoidable arsenic mobilization during leaching (Ling et al., 2021; Zhang et al., 2023; Wang et al., 2021).

Beyond process-level considerations, a growing body of environmental and geochemical literature shows that arsenic management in antimony hydrometallurgy cannot be evaluated solely by removal efficiency but must also account for the long-term stability and mobility of As-bearing residues. Field and laboratory studies demonstrate that arsenic and antimony often exhibit coupled behavior in soils, tailings, and mining-impacted environments, with redox conditions, mineral transformations, and aging processes firmly controlling their mobility (Verbeeck et al., 2021; Drahota et al., 2023; Radková et al., 2023). Recent investigations further show that poorly stabilized arsenic phases may remobilize over time, particularly under changing pH–Eh conditions, effectively shifting environmental risk from liquid effluents to solid residues (Zhao et al., 2022; Zhou et al., 2024). These findings reinforce the need for hydrometallurgical flowsheets to prioritize the formation of thermodynamically stable arsenic phases (e.g., scorodite-type structures) and to critically assess residue stability as part of environmental, health, and safety (EHS) evaluation, rather than treating arsenic precipitation as a purely operational endpoint (Tian et al., 2020).

The literature reports two dominant strategies for arsenic management: (i) stabilization as arsenates or scorodite-like phases, and (ii) co-precipitation into mixed sludges.

Scorodite-based stabilization is widely regarded as chemically stable under controlled conditions, but it requires strict pH, redox, and iron control and is sensitive to long-term environmental exposure (Drahota et al., 2023; Radková et al., 2023). In contrast, co-precipitation into amorphous sludges is operationally simpler but often yields poorly characterized residues with uncertain long-term stability, effectively transferring arsenic from solution to solid waste without resolving its environmental risk (Ling et al., 2024; Zhao et al., 2022).

Several authors explicitly caution against flowsheets that report high Sb recovery while relegating arsenic to secondary residues without a stability assessment, noting that such approaches may be incompatible with modern regulatory frameworks (Seal, 2021; Periferakis et al., 2022).

Arsenic management must be evaluated as a lifecycle issue rather than a unit operation. Processes that merely shift arsenic from the liquid to the solid phase without demonstrating long-term stability risk undermining the sustainability claims of antimony recovery routes.

Across the reviewed literature, residues and effluents consistently emerge as the weakest link in antimony hydrometallurgical flowsheets. Sulfide-bearing effluents, chloride-rich circuits, and arsenic stabilization challenges are frequently acknowledged yet rarely quantified or integrated into holistic process evaluations. As a result, many proposed routes achieve impressive laboratory-scale performance while remaining environmentally fragile.

This section reinforces a central conclusion of the review: the true measure of technological readiness in antimony hydrometallurgy is not extraction yield but the ability to manage residues and effluents safely, predictably, and in compliance with environmental regulations.

Therefore, the following section synthesizes the critical findings of this review, identifies unresolved research gaps, and outlines future directions for integrated, EHS-compatible antimony processing flowsheets.

9. Research Gaps and Future Perspectives

Despite significant progress in antimony hydrometallurgy between 2020 and 2025, the literature reveals persistent structural gaps that hinder the translation of laboratory-scale advances into robust, industrially deployable flowsheets. Across most studies, improvements in leaching efficiency are reported in isolation, while pretreatment design, impurity stabilization, effluent management, and final product specification remain poorly integrated. As a result, many proposed routes exhibit favorable extraction performance but low overall technology readiness when evaluated from environmental, operational, and circularity-oriented perspectives. This mismatch is particularly critical given antimony’s growing strategic relevance, supply concentration, and vulnerability within global trade networks (Bussolesi et al., 2024; Kanellopoulos et al., 2024; Khorshidi et al., 2025; Zhao G. et al., 2023).

A recurring research gap concerns the misalignment between chemical performance and system-level feasibility. Highly aggressive leaching systems—particularly acid oxidative and chloride-based routes—often achieve excellent Sb dissolution but impose substantial downstream purification burdens and generate complex effluents that challenge long-term environmental compliance. Conversely, more selective systems, such as alkaline sulfide leaching, show higher practical relevance but remain constrained by kinetic limitations, sulfide management, and residue stability. These trade-offs are rarely quantified in a comparative or lifecycle-oriented manner, leading to an overestimation of process maturity.

Another critical gap concerns the treatment of arsenic, bismuth, and other harmful impurities. Although many studies report effective removal or coprecipitation, far fewer assess the long-term stability, remobilization risk, or regulatory acceptability of the resulting residues. Environmental and geochemical evidence increasingly shows that inadequate stabilization strategies may merely shift risk from liquid effluents to solid waste streams, undermining the sustainability claims of otherwise efficient hydrometallurgical routes. Future research must therefore move beyond short-term removal metrics and incorporate residue aging, speciation stability, and EHS performance as core evaluation criteria.

From a circular-economy standpoint, a substantial portion of the literature still conflates contaminant removal with resource recovery. Adsorption, ion exchange, and polishing technologies are often presented as successful solutions, despite the lack of regeneration, Sb product isolation, or demonstrable integration into closed-loop flowsheets. This distinction is particularly relevant for secondary resources, where true valorization requires not only Sb extraction but also the production of marketable products and the minimization of secondary waste generation.

To synthesize the interdependencies among process maturity, environmental risk, and circularity potential, Figure 12 places the main antimony hydrometallurgical routes within a TRF × EHS × circularity framework.

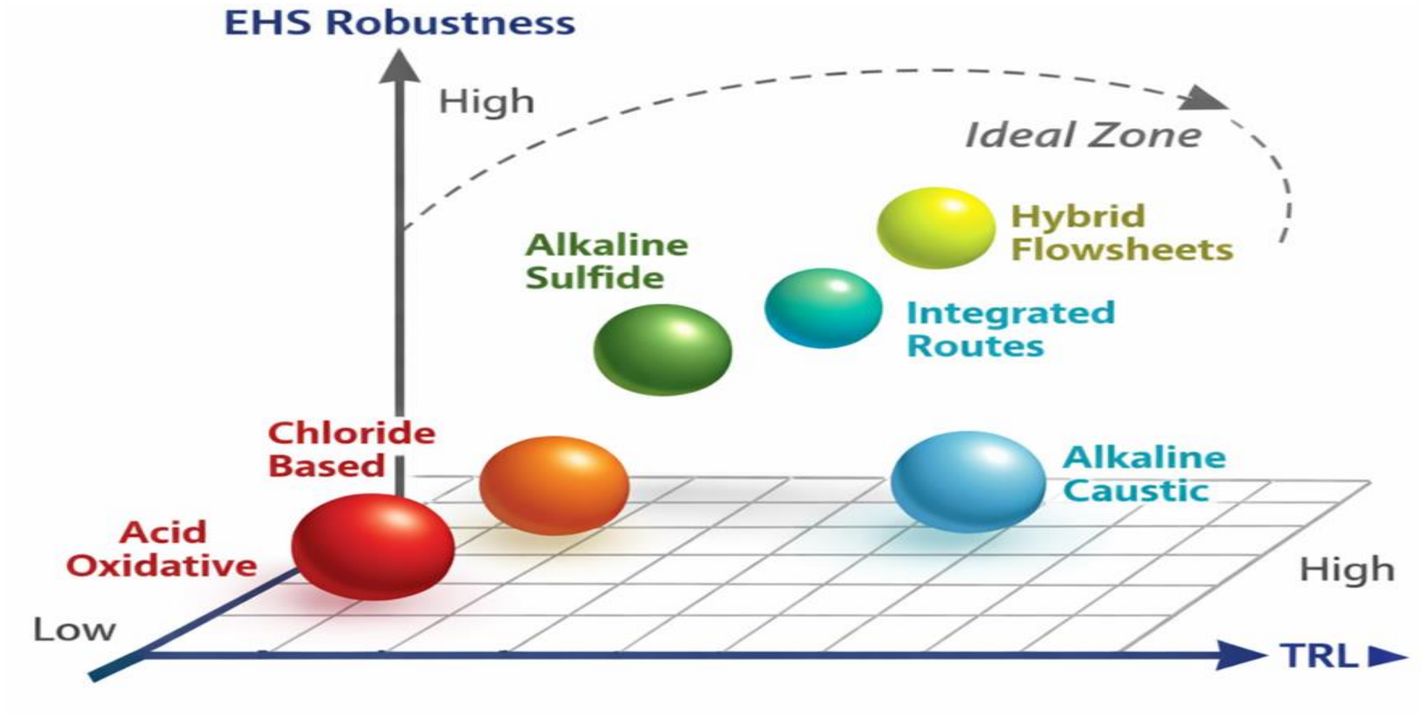

Figure 12. TRF × EHS × Circularity critical positioning map for antimony hydrometallurgical routes. Adapted from concepts and comparative assessments reported in Dembele et al. (2022, 2024, 2025), Ling et al. (2021, 2024), Alguacil (2025), Panayotova and Panayotov (2025).

As shown in Figure 12, no single processing route currently occupies the optimal region that combines high TRF, strong EHS performance, and genuine circularity. Alkaline sulfide leaching appears closest to industrial relevance for sulfide-dominated feeds, yet remains constrained by sulfide-handling and kinetic limitations. Acidic and chloride-based systems demonstrate high chemical power but score poorly on EHS robustness unless paired with advanced purification and effluent treatment. Emerging hybrid flowsheets—integrating selective pretreatment, moderate leaching chemistry, and regenerative purification—show the greatest potential to move toward the upper-right quadrant of the map, although their maturity remains limited.

Looking ahead, future advances in antimony hydrometallurgy are unlikely to come from incremental improvements in leaching reagents alone. Instead, progress will depend on holistic process concepts that explicitly couple mineralogical understanding, pretreatment strategy, selective leaching, purification logic, and residue stabilization within a unified design framework. Research efforts that integrate TRF, EHS, and circularity considerations from the outset—rather than as post hoc evaluations—are most likely to deliver scalable, sustainable, and industrially credible solutions for antimony recovery.

10. Conclusions

This review demonstrates that the principal challenges in antimony hydrometallurgy are not rooted in dissolution chemistry, but rather in the management of impurity-rich systems and the integration of individual unit operations into coherent and scalable flowsheets. Pretreatment strategies play a critical enabling role by modifying mineralogical accessibility and speciation; however, their contribution must be evaluated in terms of downstream implications rather than isolated performance metrics.

Leaching systems across acidic, alkaline, and oxidative routes routinely achieve high Sb extraction efficiencies, yet such results often obscure the generation of chemically complex pregnant solutions containing arsenic, iron, lead, bismuth, and other problematic species. Consequently, leaching efficiency alone provides limited insight into process viability when divorced from purification performance.

Purification and separation stages ultimately determine product quality, environmental compliance, and technology readiness, emerging as the most failure-prone components of Sb hydrometallurgical flowsheets. While numerous solvent extraction, adsorption, precipitation, and membrane-based approaches have been proposed, many remain insufficiently validated at the integrated process level, with limited consideration of reagent recycling, impurity accumulation, and long-term operational stability.

Overall, the literature reveals a persistent imbalance between detailed unit-operation studies and scarce flowsheet-level validation. Bridging this gap represents the primary challenge for advancing antimony hydrometallurgy from laboratory exploration to industrially credible and sustainable practice. Future efforts should emphasize impurity-aware process design, coupled evaluation of leaching and purification stages, and explicit assessment of technology readiness to support realistic scale-up and deployment.

11. Declarations

Author Contributions

Antonio Clareti Pereira: Conceptualization, methodology, literature review, data curation, critical analysis, visualization, writing—original draft, writing—review and editing.

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All data analyzed in this study are derived from published literature sources cited in the reference list. No new experimental dataweregenerated.

Conflicts of Interest

The author declares no conflict of interest.

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