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Integrated Process Routes for Spent Pot Lining Valorization: From Hazard Detoxification to Closed-Loop Recovery of Fluorides, Carbon, and Refractories

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ABSTRACT **Review Article**

Spent pot lining (SPL) is simultaneously a hazardous residue and a heterogeneous secondary resource containing carbon, fluoride-bearing electrolyte, sodium, aluminum, refractory minerals, and reactive or cyanide-bearing species. This structured critical narrative review reframes SPL management as a flowsheet-integration problem rather than a competition among isolated physical, hydrometallurgical, thermal, or co-processing technologies. A fixed core evidence corpus of 106 publications from January 2020 through 19 August 2026, supplemented only by three pre-2020 foundational or regulatory sources excluded from corpus counts, is assessed according to feed fraction, hazard destruction, elemental recovery, reagent and water demand, energy intensity, product quality, secondary residues, evidence scale, and industrial integration. Eight integrated process architectures are compared, ranging from physical separation plus selective hydrometallurgy to thermal-hydrometallurgical hybrids, direct co-processing, closed-loop aluminum-smelter recycling, and a near-zero-waste SPL-refinery concept. The central finding is that high extraction alone is an insufficient measure of circularity: carbon, fluorine, sodium, aluminum, and cyanide-bearing species must be tracked to qualified products, recycle streams, controlled emissions, or final residues. Carbon-rich first-cut SPL generally favors selective defluorination followed by carbon upgrading, whereas refractory-rich second cut requires mineral-oriented recovery or controlled co-processing. Thermal steps are most defensible when they solve a defined hazard or separation constraint and when condensates and off-gases are deliberately recovered. Hydrometallurgical routes offer stronger control over fluoride recovery but can shift burdens to reagent consumption, saline liquors, and water treatment. The most promising long-term architecture is feed-adapted and hybrid, but the evidence base remains laboratory-dominant. Research priorities are representative pilot campaigns, full constituent balances, product qualification, recycle-loop stability, and coupled techno-economic and life-cycle assessment.

Keywords: Spent Pot Lining, Aluminum Smelting, Fluoride Recovery, Carbon Recovery, Process Integration, Circular Economy.

Highlights

- Integrated SPL routes provide a stronger basis for decision-making than technology-by-technology comparisons.
- The elemental fate of C, F, Na, and Al determines whether treatment achieves genuine resource recovery.
- Detoxification, immobilization, recovery, and recycling are fundamentally different process outcomes.
- Feed-adapted hybrid routes offer the strongest pathway toward product-qualified, closed-loop SPL management.

The graphical abstract summarizes the process logic adopted throughout this review: SPL is first segregated and characterized, then routed through complementary physical, hydrometallurgical, thermal, precipitation, and purification modules, while the fate of C, F, Na, Al, cyanide-bearing species, and refractory constituents is explicitly controlled.

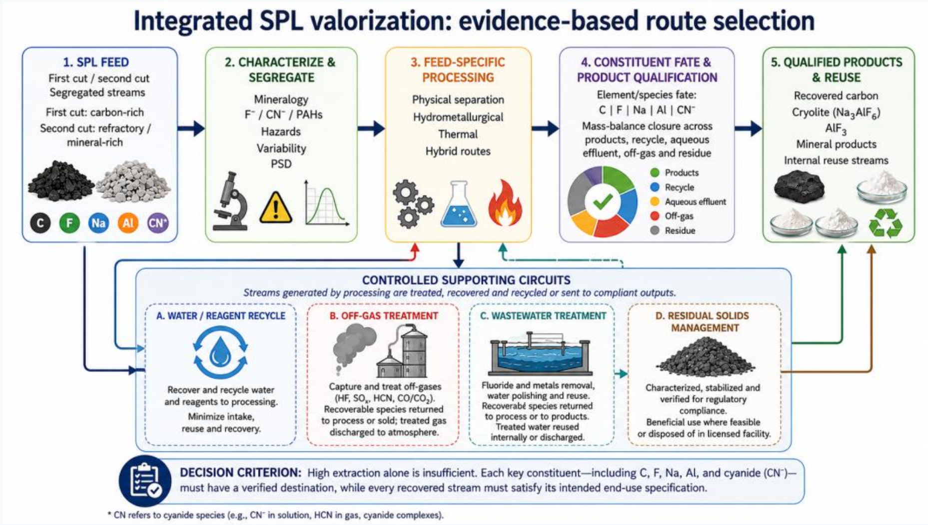

Figure GA. Graphical abstract: integrated SPL valorization from selective preparation to closed-loop reuse.

Note. Original conceptual synthesis developed for this review from the process-integration logic evaluated in the literature corpus.

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Introduction

Spent Pot Lining Generation in Primary Aluminum Production

Spent pot lining is generated when the cathodic and insulating lining of a Hall-Héroult reduction cell reaches the end of its service life and is removed during relining. The material is intrinsically stratified: the first cut is dominated by carbonaceous cathode blocks and ramming materials impregnated by electrolyte, whereas the second cut contains a larger fraction of refractory and insulating materials. The literature increasingly treats these fractions as distinct secondary resources rather than as a single waste stream, because their carbon content, mineralogy, fluoride speciation, and downstream value propositions differ substantially (Chen, Li, Bu, Chehreh Chelgani, et al., 2022; Dong et al., 2023; Khan et al., 2026; Kitchamsetti et al., 2026; Liu, 2020; Zhou et al., 2024).

The practical consequence is that SPL composition is created by cell design, bath chemistry, penetration of sodium and fluoride-bearing electrolyte into the lining, cathode wear, local temperature gradients, dismantling practice, and the degree to which carbon and refractory layers are kept separate. This variability explains why treatment results obtained with one SPL source cannot be transferred mechanically to another. Industrial guidance and systemic accounting studies therefore emphasize site-specific characterization and integration with the wider aluminum-smelter material system (Billy et al., 2022; Nunez, 2020; Pereira, 2025b).

Why SPL Is More Than a Waste-Disposal Problem

The hazard classification of SPL is closely associated with mobile fluoride and cyanide-bearing species, but the same matrix also contains potentially recoverable carbon, sodium and aluminum in electrolyte phases, and refractory minerals. Recent reviews have consequently shifted from disposal-oriented language toward resource utilization, carbon recovery, fluoride detoxification, and circular use (Agrawal et al., 2021; Chen, Li, Bu, Chehreh Chelgani, et al., 2022; Dong et al., 2023; Khan et al., 2026; Ma et al., 2024b; Pereira, 2025a). The central engineering question is not whether SPL can be treated, but whether a treatment sequence can simultaneously control hazards, concentrate value, produce specification-compliant products, and avoid creating a secondary liquid, gaseous, or solid waste burden.

This distinction matters because an apparently high 'removal' efficiency may simply transfer fluoride from a solid into an untreated liquor, or convert soluble fluoride into an immobilized phase without recovering it as a product. Likewise, destroying cyanide thermally may improve safety while consuming carbon that could otherwise have been recovered. Conversely, aggressive chemical purification can preserve carbon but impose high reagent demand and saline effluent management. The literature contains examples of each trade-off, including leaching-based carbon purification, thermal detoxification, low-pressure electrolyte removal, co-processing, and product-oriented precipitation (Birry & Gauthier, 2024; Z. Chen et al., 2022; Dzikunu et al., 2023; Ma et al., 2024a; Sommerseth et al., 2025; Yang et al., 2022).

Environmental and Operational Hazards

SPL processing combines chemical and process-safety hazards. Fluoride may occur in water-soluble salts, cryolitic phases, NaF, CaF_2, and more strongly bound mineral forms; cyanide may be present in free or complexed forms; and strongly alkaline material may coexist with metallic Al, Al_4C_3, and AlN. Contact with water or acidic solutions can therefore create gas-evolution risks involving H_2, CH_4, NH_3, and, under unfavorable pH conditions, HCN. Thermal processing introduces a different set of concerns: fluoride-bearing particulate, volatile fluorides, dust, and condensates must be contained and captured (Han et al., 2023; Lin et al., 2023; Liu, 2020; Sang, Liang, et al., 2022; Su et al., 2022; Zhang et al., 2020).

Accordingly, storage, crushing, grinding, leaching, drying, and thermal treatment should be designed as one safety envelope. The safest route is not necessarily the one with the smallest number of unit operations; it is the route in which hazardous species are predictably transformed and their fate is verified by mass balance. This systems view is consistent with recent work on fluoride prevention and control, cyanide destruction, and thermal emission management (Han et al., 2023; Lin et al., 2023; Song et al., 2026; Su et al., 2022).

Limitations of Conventional SPL Reviews and Objective of This Review

Most conventional classifications separate SPL technologies into physical, hydrometallurgical, thermal, reuse, and disposal categories (Chen, Li, Bu, Chehreh Chelgani, et al., 2022; Dong et al., 2023; Khan et al., 2026; Ma et al., 2024b; Pereira, 2025a; Pereira, 2025b; Zhou et al., 2024). That taxonomy is useful for cataloguing unit operations but weak for industrial decision-making because a viable plant normally combines several categories. A carbon-recovery flowsheet, for example, may require selective dismantling, crushing, washing or leaching, solid-liquid separation, wastewater treatment, drying, thermal polishing, gas treatment, and product qualification. The performance of the complete chain can differ materially from the apparent performance of the headline unit operation.

The objective of this review is therefore to critically evaluate integrated SPL treatment and valorization routes by tracking the fate of carbon, fluorine, sodium, aluminum, cyanide-bearing species, and refractory constituents from feed preparation to final products and residues. The novelty lies in comparing process architectures rather than isolated technologies and in using constituent-fate, product-quality, recycle-loop, safety, and scale-up criteria as common decision variables.

Review Methodology and Critical Assessment Framework

Literature-Search Strategy and Evidence Corpus

The review was designed as a structured critical narrative review with a fixed core evidence window from 1 January 2020 through 19 August 2026. The final core corpus contains 106 unique publications. Coverage was re-audited using the combined concepts ("spent pot lining" OR "spent potlining" OR "spent pot liner" OR "spent cathode carbon" OR "overhaul slag") AND (fluoride OR cyanide OR carbon OR graphite OR refractory OR leach* OR roast* OR vacuum OR flotation OR precipitation OR co-processing OR recycling OR valorization), together with backward and forward citation checking and publisher-record verification. Eligible records were peer-reviewed journal papers or substantive conference contributions that reported SPL-specific experimental, process, material-flow, environmental, or industrial evidence; recent reviews were retained only for contextual synthesis. Preprints, duplicate records, papers centered on other aluminum wastes without separable SPL evidence, and records lacking sufficient methodological or process information were excluded.

The working bibliography had been assembled iteratively before the present re-audit, and raw pre-deduplication database exports were not retained. A retrospective PRISMA-style identification count would therefore be artificial and is deliberately not reconstructed. Instead, transparency is provided through explicit eligibility rules, a fixed 106-study core corpus, and study-level coding in Supplementary Table S1. Three older sources are used only for foundational characterization, historical cyanide-treatment context, and regulation; they are excluded from all 2020-2026 corpus counts (Courbariaux et al., 2004; Ospina & Hassan, 2017; United States Environmental Protection Agency [U.S. EPA], 2000). The core corpus spans physical separation, hydrometallurgy, precipitation, thermal and vacuum treatments, process intensification, gasification/combustion, co-processing, product recovery, and industrially oriented SPL management.

Evidence Classification

To prevent laboratory extraction data from being interpreted as industrial feasibility, each empirical or implementation-oriented paper was assigned the highest explicitly demonstrated evidence level: Level 1, laboratory; Level 2, bench; Level 3, pilot; Level 4, demonstration or industrial-host trial; and Level 5, sustained industrial practice. Reviews, modeling papers without process demonstration, guidance, and system-accounting studies were retained as contextual evidence but were not assigned a process-performance scale. Coding was intentionally conservative: scale was not inferred from equipment descriptions when treated mass, operating mode, duration, or industrial setting was not explicit.

Table 1 operationalizes the hierarchy and reports the corpus distribution. Of the 106 core publications, 90 provide empirical or implementation evidence that could be scale-coded and 16 serve primarily contextual, review, modeling, guidance, or system-accounting roles. The resulting distribution is strongly laboratory-dominant, which constrains claims about continuous operability, recycle stability, corrosion, off-gas reliability, product acceptance, and cost.

Table 1. Evidence hierarchy, operational definitions, and distribution of the 106-study core corpus

Evidence category	Operational definition used in this review	Claims supported	Core records, n
Level 1 - Laboratory	Batch or small-apparatus proof of chemistry, mechanism, separation, detoxification, or product formation.	Mechanism, selectivity, kinetics, preliminary product quality.	85
Level 2 - Bench	Integrated kilogram-scale batch or semi-integrated equipment with explicit treated mass and process controls.	Preliminary operability, heat/mass-transfer and handling constraints.	1
Level 3 - Pilot	Representative semi-continuous or continuous pilot campaign with integrated recycle/separation.	Scale-up behavior, recycle response, preliminary reliability.	0
Level 4 - Demonstration	Industrial-host or near-commercial trial under representative operating conditions.	Host-process compatibility, emissions/product effects, operating integration.	2
Level 5 - Industrial practice	Sustained site or commercial practice with an identified treatment/reuse outlet.	Demonstrated integration and practical viability within the reported boundary.	2
Contextual / non-scale	Review, modeling without process demonstration, guidance, or system/material-flow accounting.	Context, mechanism framing, environmental or management interpretation; not independent proof of process scale.	16

Note. The level is the highest scale explicitly supported by each core publication; no scale was inferred where the paper did not report sufficient evidence. Counts sum to 106. The hierarchy is used to separate chemical feasibility from evidence of sustained industrial operability. Supplementary Table S1 provides the study-level coding.

The critical rule applied throughout is that detoxification, immobilization, recovery, and recycling are not synonyms. Detoxification reduces hazard; immobilization reduces mobility; recovery transfers a constituent into a separable product stream; recycling requires a qualified use that displaces a primary material. A route is scored more favorably only when it advances along this chain without imposing disproportionate burdens elsewhere in the flowsheet.

Data Extraction, Route Assignment, and Critical Synthesis

For each core publication, the review extracted feed identity (first cut, second cut, spent cathode carbon, mixed SPL, or treated derivative), principal unit operations, dominant hazard-control function, C/F/Na/Al fate, reported recovery or product-quality endpoints, utility and secondary-stream information when available, evidence scale, and stated industrial setting. The critical comparison used nine recurring dimensions: feed representativeness, hazard control, constituent mass balance, product qualification, reagent/water/energy demand, secondary pollution, scale-up evidence, recycle/integration behavior, and techno-economic or life-cycle information. Missing data were retained as evidence gaps rather than scored as favorable performance.

The eight route architectures were derived from the sequence and dominant function of reported unit operations rather than from technology labels alone. A study could support more than one architecture, but one primary route was assigned in Supplementary Table S1 for traceability. Section 6 reports non-mutually-exclusive anchor-study counts for each architecture and the highest explicit evidence level among those anchors. The qualitative route matrix in Section 12 uses an explicit rating rubric and a separate evidence-confidence descriptor so that process potential is not conflated with maturity.

SPL as a Process Feed: Composition, Mineralogy, Reactivity, and Variability

First Cut, Second Cut, and the Value of Selective Dismantling

The first cut is generally carbon-rich and therefore the principal target for graphite or carbon recovery, while the second cut contains refractory and insulating materials that are better suited to mineral recovery, conversion, or co-processing. Recent work on recovered graphite, high-temperature cleaning, and refractory recycling reinforces the benefit of preserving this distinction (Angelopoulos et al., 2021; Hou et al., 2021; Kitchamsetti et al., 2026; X. Li et al., 2025; Merchichi et al., 2026; Robshaw et al., 2022; Sommerseth et al., 2025). Mixing the fractions before treatment dilutes carbon, increases acid or alkali consumption, broadens mineralogy, and can make a single purification route unnecessarily severe.

Figure 1 illustrates how the first and second cuts differ and why the distribution of value-bearing and hazardous phases should be considered before selecting a treatment route.

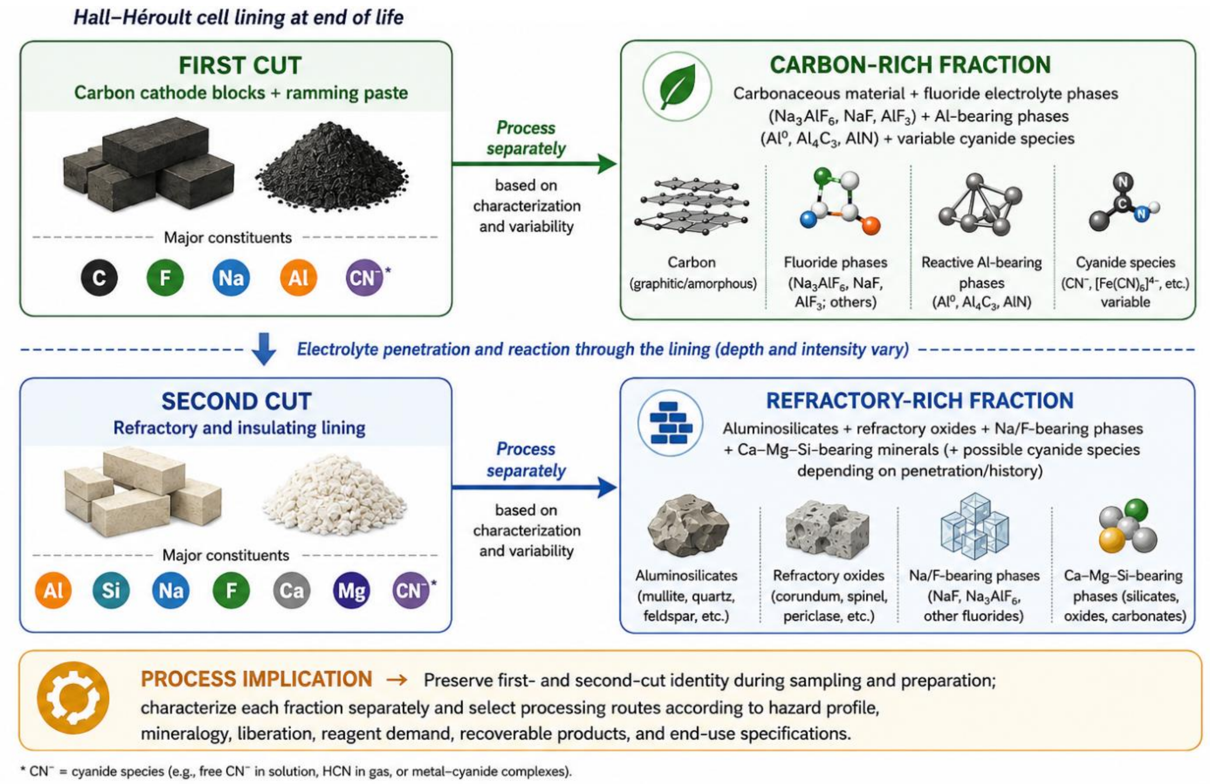

Figure 1. Formation of first- and second-cut SPL and the distribution of value-bearing and hazardous phases.

Note. Conceptual synthesis adapted from recent SPL characterization, recycling, and end-of-life treatment literature (Chen, Li, Bu, Chehreh Chelgani, et al., 2022; Dong et al., 2023; Liu, 2020; Sommerseth et al., 2025).

Carbon, Fluorine, Sodium, and Aluminum Speciation

Carbon in the first cut can retain a graphitic structure with varying amounts of ash, electrolyte, aluminosilicate, and metallic or carbide/nitride contaminants. This explains why carbon 'recovery' requires both liberation and decontamination; flotation can reject some mineral phases, but deeply penetrated electrolyte usually demands chemical or thermal treatment (Angelopoulos et al., 2021; Chen et al., 2024; Chen, Li, Bu, Wang, et al., 2022; Li et al., 2022; Li, Chen, Chen, et al., 2025; Li, Chen, Ouyang, et al., 2025; Merchichi et al., 2026; Sundby et al., 2020; Yao, Zhong, Xiao, Ye, Tang, & Wang, 2021; Yuan, Ding, & Li, 2022; Zhou et al., 2023). High-value applications such as battery or anode feedstock impose much tighter impurity control than use as fuel or reductant (Robshaw et al., 2022; Yan et al., 2026; Yang et al., 2020; Zheng et al., 2024).

Fluorine is distributed among readily soluble salts, cryolite-related phases, NaF, CaF_2, and more refractory or strongly bound compounds. This distribution controls whether water washing is sufficient, whether caustic or acid chemistry is needed, and whether roasting is used to transform poorly soluble phases before leaching (Dong, Jiao, Liu, & Ling, 2025; Dong, Jiao, Liu, & Zhang, 2025; Lan et al., 2024; Nemchinova et al., 2022; Nie et al., 2020; Tang et al., 2025; Tropenauer et al., 2021; Xu et al., 2023; Xu et al., 2024; W. Yang et al., 2024; Zhang et al., 2025; Zhu et al., 2023; Zhu et al., 2026). Sodium and aluminum are coupled in the same mineral transformations; therefore, a route optimized solely for total F extraction can produce Na-rich solutions or Al-bearing precipitates that require their own purge or recovery strategy.

Cyanide Chemistry and Water-Reactive Phases

Cyanide-bearing species are central to SPL hazard management, but their importance extends beyond compliance, as the sequence of wet and thermal operations can alter exposure risk. Oxidative, microwave, calcification, and co-processing studies demonstrate that cyanide destruction can be rapid under suitable thermal conditions, yet fluoride behavior under the same conditions may involve volatilization, fixation, or transfer to ash (Z. Chen et al., 2022; Sang, Liang, et al., 2022; Sang, Liu, et al., 2022; Song et al., 2026; Su et al., 2022; Zhang et al., 2020). Thus, a thermal step selected for cyanide destruction must be evaluated simultaneously for F fate and off-gas control.

Reactive metallic Al, Al_4C_3, and AlN create an additional constraint for wet front ends. In process-design terms, these phases mean that 'water washing' is not merely a benign washing operation: it may be a gas-generating reaction stage requiring ventilation, monitoring, controlled addition, and an operating philosophy that prevents accumulation of combustible or toxic gases. This is one reason why representative feed characterization is a safety requirement rather than an analytical refinement.

Table 2 consolidates the principal SPL constituents, representative levels explicitly reported in the literature, their process relevance, and the preferred separation or control response. Reported values are treated as descriptive envelopes or study-specific examples rather than universal feed specifications.

Table 2. SPL composition, representative reported levels, hazardous constituents, and recovery targets

Constituent/feature	Typical occurrence and representative reported level	Primary concern/value	Preferred process response
Graphitic/amorphous carbon	Predominantly first cut; representative first-cut reports are commonly carbon-dominant, with values around 55-65 wt% in characterized feeds.	Major recoverable mass and potential carbon/graphite product.	Preserve by selective dismantling; remove electrolyte/mineral ash to the level required by the target use.
NaF / soluble salts	Both cuts in electrolyte-impregnated regions; one characterized first-cut feed reported NaF near 15 wt%.	Soluble F and Na; water treatment and recycling burden.	Controlled washing; crystallization/precipitation; recycle with defined purge.
Cryolite-related phases	First cut and contaminated refractory; abundance is source-specific and coupled to Na-Al-F inventory.	Recoverable electrolyte value but only partial direct solubility.	Alkaline/acid transformation, roasting-leaching, vacuum separation, or product-oriented recovery.
CaF_2 / bound fluoride	Mineral/refractory fraction; generally a persistent residual fluoride phase rather than a universal fixed fraction.	Difficult defluorination; residual leachability.	Acid/hydrothermal treatment, mineral transformation, or intentional stable mineral product.
Cyanide-bearing species	Both cuts and highly variable; values approaching about 1 wt% have been reported for some SPL materials.	Hazard and possible HCN risk during inappropriate wet/acid conditions.	Verified destruction/oxidation; pH, ventilation, and gas control.
Reactive Al phases (Al, Al_4C_3, AlN)	Local and strongly source-dependent; often not quantified on a common basis.	Potential H_2, CH_4, and NH_3 generation on wetting.	Characterize before wet processing; controlled conditioning and gas capture.
Aluminosilicates/oxides / refractory minerals	Predominantly second cut; mineral fraction dominates after selective removal of carbonaceous first cut.	Mineral value, acid consumption, and product-performance effects.	Physical separation, mineral conversion, or controlled co-processing.
Na/Al-rich refractory phases	Second cut; source-specific Na-Al-bearing mineral inventory.	Potential salt/mineral recovery and alkali burden.	Selective leaching or high-temperature conversion with closed Na/Al accounting.

Note. Reported levels are intentionally descriptive rather than pooled means because SPL composition depends on cell design, bath penetration, service history, dismantling, and fraction definition. Compiled and adapted from foundational and recent characterization, defluorination, and recycling studies (Courbariaux et al., 2004; Ospina & Hassan, 2017; Wang et al., 2020; Liu, 2020; Dong et al., 2023; Dzikunu et al., 2023; X. Li et al., 2025).

The key implication is feed-specificity. A carbon-rich first cut with moderate electrolyte contamination should not be forced through the same flowsheet as a mixed, refractory-rich SPL with high soluble fluoride or high cyanide. The feed itself is the first route-selection variable.

Front-End Processing and Feed Preparation

Selective Dismantling, Crushing, and Size Classification

Front-end preparation determines the downstream selectivity available. Selective dismantling preserves the intrinsic separation between carbon-rich and refractory-rich layers; crushing then exposes electrolyte-rich pores and liberates mineral attachments. However, progressively finer grinding increases energy demand, dust generation, and the surface area available for rapid reactions during wet processing. Flotation studies demonstrate that physical separation can exploit differences between graphite and mineral matter, whereas recent thermal and low-pressure work shows that relatively coarse first-cut particles can also be treated when volatilization rather than fine-particle dissolution is the governing mechanism (Angelopoulos et al., 2021; Chen et al., 2024; Sommerseth et al., 2025).

The optimal particle size is therefore route-dependent. Hydrometallurgy often benefits from improved liberation and reduced diffusion length; flotation requires an appropriate liberation-size window; thermal vacuum or low-pressure treatment can avoid excessive milling but may suffer incomplete purification of larger pieces; and direct co-processing may tolerate a broader size range while imposing feeding and combustion constraints (Angelopoulos et al., 2021; Dzikunu et al., 2023; Sommerseth et al., 2025; Wang et al., 2023; Xin et al., 2022). A universal grinding specification is consequently inappropriate.

Washing, Conditioning, Blending, and Homogenization

Water washing is attractive for removing soluble salts before more selective stages, and it has been investigated both as a stand-alone treatment and as the first step of successive-leaching routes (Dzikunu et al., 2023; X. Li et al., 2025; Shirmahd et al., 2023; Wang et al., 2023). Its weakness is that dissolved F and Na become a water-management problem unless the liquor is deliberately processed for product recovery or stable recycle. Washing can also activate water-reactive phases. For this reason, wash-water chemistry, gas generation, residence time, and counter-current recycle should be part of the flowsheet definition rather than omitted as ancillary details.

Blending and homogenization reduce short-term feed variability but can also destroy useful segregation if first and second cuts are mixed indiscriminately. The preferred philosophy is hierarchical: segregate by material class first, homogenize within each class second, and only then blend to a controlled process specification. This is consistent with the broader move toward material-flow accounting and source-specific recovery strategies (Billy et al., 2022; Lin et al., 2023; Nunez, 2020).

Table 3 summarizes how front-end decisions influence downstream performance. The table makes explicit that preparation is not neutral: each operation changes both selectivity and risk.

Table 3. Effects of front-end preparation on downstream SPL treatment

Front-end operation	Primary benefit	Potential penalty	Best fit
Selective dismantling	Preserves carbon/refractory separation	Requires disciplined demolition and handling	All integrated routes
Coarse crushing	Enables handling and exposes penetrated electrolyte	Dust; incomplete liberation	Thermal/vacuum and co-processing routes
Fine grinding	Improves liberation and leach kinetics	Energy, dust, filtration difficulty	Selective leaching and flotation
Screening/classification	Creates size-specific treatment windows	Additional equipment and recirculating load	Hybrid physical-chemical routes
Froth flotation	Rejects part of mineral matter; upgrades graphite	Needs liberated particles and water circuit	Carbon-oriented first-cut routes
Water washing	Removes soluble F/Na and conditions feed	Creates saline liquor; gas-evolution risk	Hydrometallurgical and hybrid routes
Feed homogenization	Stabilizes reagent and heat demand	Can mask mineralogical variability	After first-/second-cut segregation

Note. Adapted from physical-separation, selective-leaching, water-leaching, ultrasonic, and low-pressure thermal studies (Angelopoulos et al., 2021; Chen et al., 2024; Dzikunu et al., 2023; Shirmahd et al., 2023; Sommerseth et al., 2025; Tian et al., 2025; Wang et al., 2023).

Process Modules Available for SPL Treatment

Figure 2 reorganizes the main unit-operation families into a staged technology toolbox. The redesign separates front-end preparation, separation/conversion, and product/reuse functions so that complementary modules can be read without implying that every module must be connected directly to the feed.

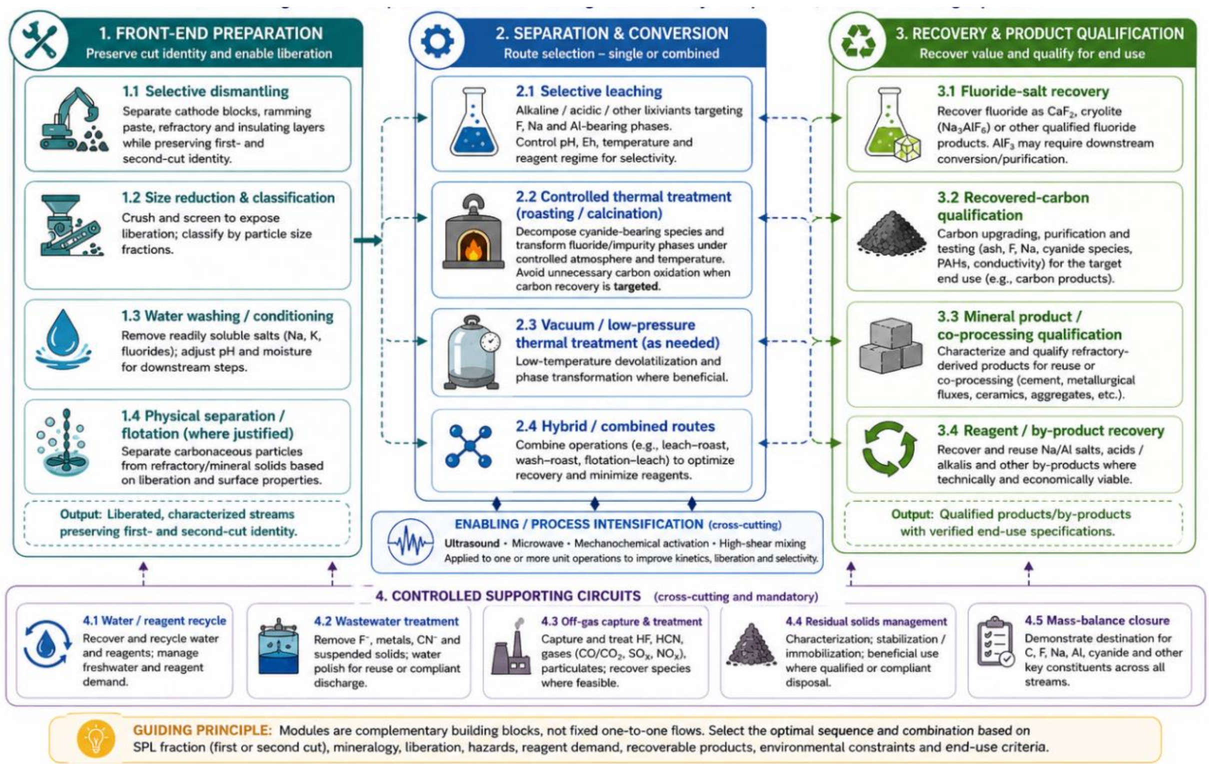

Figure 2. Technology toolbox for constructing integrated SPL treatment routes.

Note. Original synthesis adapted from the hydrometallurgical, thermal, physical-separation, and co-processing literature (Chen, Li, Bu, Chehreh Chelgani, et al., 2022; Dong et al., 2023; Khan et al., 2026; Ma et al., 2024b; Pereira, 2025a; Zhou et al., 2024).

Water, Alkaline, Acid, and Sequential Selective Leaching

Hydrometallurgical treatment offers the most direct control over the transfer of fluoride, sodium, and aluminum from SPL into separable aqueous streams. Water washing removes readily soluble salts; alkaline systems can attack cryolitic or Al-bearing phases; and acid systems can dissolve or transform fluoride-bearing minerals that are poorly responsive to water alone (Nemchinova et al., 2022; Nie et al., 2020; Robshaw et al., 2020; Shirmahd et al., 2023; Tropenauer et al., 2021; Xu et al., 2023; Xu et al., 2024; Yuan, Li, & Ding, 2022). The strongest hydrometallurgical concepts use staged chemistry rather than a single aggressive leach, because sequential treatment can exploit differences in solubility and reduce unnecessary dissolution of carbon or refractory gangue.

Successive selective leaching illustrates this logic: soluble salts are removed first, followed by targeted alkaline or acid dissolution and then solid-liquid separation, washing, and product recovery (Dzikunu et al., 2023; Sathish et al., 2025; Yang et al., 2022; Yuan, Ding, & Li, 2022). Ultrasound and microwave-hydrothermal assistance can intensify mass transfer or mineral transformation (Tian et al., 2025; Tian et al., 2024; Wang et al., 2023; Xu et al., 2023; Xu et al., 2024). The critical trade-off is selectivity versus reagent and water consumption. High extraction is not a sufficient outcome if the resulting liquor contains a difficult mixture of Na, Al, F, Ca, Si, and acid/alkali neutralization salts.

Precipitation and Fluoride-Product Recovery

Once fluoride is dissolved, precipitation becomes the decisive step that distinguishes wastewater treatment from resource recovery. Recent studies investigate flexible precipitation, fractional precipitation, cryolite regeneration, and AlF_3 synthesis (Dong, Jiao, Liu, & Ling, 2025; Lan et al., 2024; Ma et al., 2024a; Yang et al., 2023; Zhu et al., 2026). These routes show that fluoride-bearing liquor can be converted into saleable or internally recyclable products, but only if impurity carryover, stoichiometry, washability, and product specification are controlled.

A recurring strategic choice is whether to recover F as CaF_2, cryolite, AlF_3, or another fluoride salt. CaF_2 may offer robust immobilization and a simpler precipitation chemistry but can represent a lower-value endpoint. Cryolite and AlF_3 are more attractive for a closed aluminum loop, yet they demand tighter Na:Al:F control and lower contamination by Ca, Si, Fe, or sulfate. The most recent AlF_3 route reported very high total F leaching prior to leaching-mixture calcination, indicating a shift toward product-oriented fluoride recovery rather than disposal-oriented precipitation (Dong, Jiao, Liu, & Zhang, 2025; Lan et al., 2024; Ma et al., 2024a; Zhu et al., 2026).

Thermal Oxidation, Roasting, Microwave, and Molten-Salt Treatments

Thermal routes are chemically versatile. Conventional roasting and combustion can destroy cyanide, oxidize reactive carbon-associated phases, transform fluorides, and enable subsequent water leaching (Z. Chen et al., 2022; Deng et al., 2022; Y. Li et al., 2023; Song et al., 2026; Su et al., 2022; Zhang et al., 2023; Zhang et al., 2020; Zhu et al., 2023; Zhu, Xu, Han, Liu, Zhang, Tian, et al., 2022; Zhu, Xu, Han, Liu, Zhang, Yang, et al., 2022). Molten-salt roasting and alkali fusion can promote dissociation of fluoride-bearing phases and subsequent separation of carbon and electrolyte (Li et al., 2024; Li, Chen, Chen, et al., 2025; Li, Chen, Ouyang, et al., 2025; Lv et al., 2025; Yao et al., 2020; Yao, Zhong, Xiao, Ye, Tang, & Wang, 2021; Yuan, Ding, & Li, 2022; Zhang et al., 2025). Microwave and flash-heating approaches pursue faster heating or process intensification (Huang et al., 2022; Tian et al., 2024; Xu et al., 2023; Zhu, Xu, Han, Liu, Zhang, Tian, et al., 2022; Zhu, Xu, Han, Liu, Zhang, Yang, et al., 2022).

The weakness of thermal treatment is that 'destruction' of one hazard can create a gas-handling requirement for another constituent. Fluoride may be fixed in an ash, transferred to particulates, volatilized, or concentrated in condensates depending on temperature, pressure, mineral additives, and gas flow (Han et al., 2023; Y. Li et al., 2023; Song et al., 2026; Zhang et al., 2023; Zhang et al., 2020; Zhu et al., 2023). Consequently, the off-gas train is part of the process chemistry, not a downstream environmental add-on.

Vacuum and Low-Pressure/High-Temperature Separation

Vacuum and low-pressure thermal treatment exploits the volatility of electrolyte species relative to graphitic carbon. Thermal-vacuum studies have shown that fluoride-rich electrolyte can be removed while preserving a carbon-rich solid (Dong et al., 2024; Feng et al., 2025; W. Li et al., 2025; Sommerseth et al., 2025; Sundby et al., 2020; M. Xie et al., 2020; Xin et al., 2022). In the 2025 low-pressure heat study, 5-19 kg of first-cut batches were treated at high temperature and reduced pressure, with carbon content reaching approximately 98 wt% in the best experiment; the condensates were mainly NaF and cryolite-related material. The same work demonstrated that pressure, particle size, condensate removal, energy supply, transport distance, and condenser/filter design remain scale-up constraints (Sommerseth et al., 2025).

This route is therefore especially attractive where high-purity carbon has a credible market and where an energy-efficient, well-sealed thermal system can be integrated near a smelter. It is less compelling when the feed contains a large refractory fraction, when CaF_2 dominates the residual fluoride, or when the recovered carbon has no qualified outlet.

Direct Industrial Co-Processing and Mineral Conversion

Direct co-processing uses the carbon, mineral, or flux value of SPL in an existing high-temperature process. Cement and concrete applications are the most developed family in the recent corpus, including treated SPL as cementitious or raw-material feed (Al Jawi et al., 2020; Birry et al., 2023; Brial et al., 2021; Brial et al., 2022; Camara & Ouellet-Plamondon, 2023; Ansari et al., 2026). Other pathways include mold flux, brick kilns, asphalt, Al-Si master alloys, nepheline ceramics, copper-slag reduction, blast-furnace-slag combinations, and red-mud co-treatment (Han et al., 2024; Hou et al., 2021; Khan & Chen, 2025; M. Li et al., 2023; Y. Li et al., 2023; Lu et al., 2020; Sang, Liu, et al., 2022; Tu et al., 2023; W. Xie et al., 2020; Zhang et al., 2026; Zhang et al., 2022; Zhao, Ma, et al., 2021).

Co-processing can minimize standalone CAPEX and leverage existing heat and mineral sinks, but it is the route most vulnerable to false claims of circularity. The disappearance of SPL as a separately identifiable waste is not proof of resource recovery. A defensible co-processing route must show cyanide destruction, F partitioning among product/slag/off-gas, alkali limits, emission control, and no unacceptable degradation of the host product.

Integrated Process Routes for SPL

Figure 3 compares eight main process architectures using the modules above. Architectures are grouped by the order and dominant function of unit operations, not by specific technology names. The supporting study set includes 7 records for Route I, 9 for Route II, 8 for Routes III and IV, 15 for Route V, 12 for Route VI, and 8 each for Routes VII and VIII. These counts are non-mutually-exclusive, as studies may support multiple architectures. The figure is architectural, not prescriptive, and reports the highest evidence level among the cited anchors.

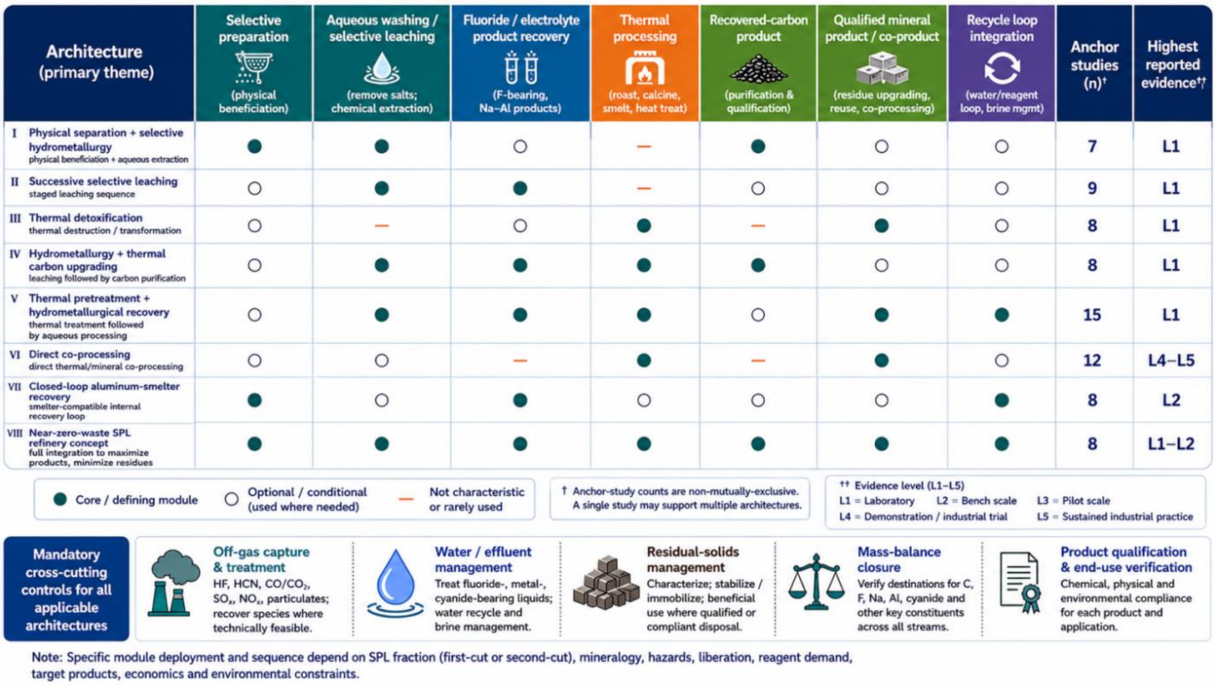

Figure 3. Comparative architecture of eight principal integrated SPL-processing routes.

Note. Original route synthesis developed for this review from the reported combinations of physical separation, leaching, precipitation, thermal treatment, carbon purification, mineral reuse, and recycle (Birry & Gauthier, 2024; Dong, Jiao, Liu, & Li, 2025; Dzikunu et al., 2023; Feng et al., 2025; Lu et al., 2020; Ma et al., 2024a; Sommerseth et al., 2025; Yang et al., 2022).

Route I - Physical Separation + Selective Hydrometallurgy

Route I starts by preserving first- and second-cut identity, followed by crushing, classification and, where beneficial, flotation or washing. A selective leach then removes F/Na/Al-bearing phases from the carbon-rich fraction; the liquor is treated for fluoride-product recovery; and the cleaned carbon and refractory-rich solids are separately qualified (Angelopoulos et al., 2021; Chen et al., 2024; Dzikunu et al., 2023; Ma et al., 2024a; Robshaw et al., 2020; Wang et al., 2023; Yang et al., 2023). Its main advantage is preservation of carbon value while maintaining chemical control over fluoride. Its disadvantages are solid-liquid separation, water demand, and the need to close or purge Na-rich liquors.

Route II - Successive Selective Leaching

Route II intensifies chemical selectivity through sequential washing and leaching. It is best suited to feeds in which soluble salts, cryolitic phases, and more refractory fluorides respond to different chemistries. Successive leaching and alkali-fusion/acid-leaching studies demonstrate the feasibility of producing a progressively cleaner carbon residue while transferring valuable species into separable solutions (Dzikunu et al., 2023; Lv et al., 2025; Sathish et al., 2025; Yang et al., 2022; Yao et al., 2020; Yao, Zhong, Xiao, Ye, Tang, & Wang, 2021; Yao, Zhong, Xiao, Ye, Tang, & Zhang, 2021; Yuan, Ding, & Li, 2022; Yuan, Li, & Ding, 2022). The route is chemically flexible but risks high reagent consumption and solution complexity if each stage is optimized independently rather than as a closed water/reagent circuit.

Route III - Thermal Detoxification

Route III prioritizes hazard destruction. Crushed SPL is blended with additives where needed and thermally treated to decompose cyanide, modify fluoride mobility, and generate a mineral residue suitable for stabilization or reuse (Z. Chen et al., 2022; Deng et al., 2022; Sang, Liang, et al., 2022; Song et al., 2026; Su et al., 2022; Zhang et al., 2020; Zhu, Xu, Han, Liu, Zhang, Tian, et al., 2022; Zhu, Xu, Han, Liu, Zhang, Yang, et al., 2022). This architecture is appropriate when immediate hazard reduction dominates the value case, but it generally underperforms product-oriented routes if fluoride is merely immobilized and carbon is combusted. It should therefore be classified as detoxification unless downstream recovery is demonstrated.

Route IV - Hydrometallurgy + Thermal Carbon Upgrading

Route IV first removes soluble and chemically accessible F/Na/Al species, then dries and thermally upgrades the carbon-rich residue. The sequence reduces the mineral load entering the high-temperature step and can make the thermal operation a polishing stage rather than the primary separation stage. Deep purification studies, combined leaching-thermal treatment, and graphite-product investigations support this architecture (Chen, Li, Bu, Wang, et al., 2022; Li et al., 2022; Li, Chen, Chen, et al., 2025; Li, Chen, Ouyang, et al., 2025; Merchichi et al., 2026; Robshaw et al., 2022; Sundby et al., 2020; Zhou et al., 2023). Its strongest use case is a carbon product with value high enough to justify multistage purification.

Route V - Thermal Pretreatment + Hydrometallurgical Recovery

Route V reverses the order: a controlled thermal step first destroys cyanide or converts difficult mineral phases, after which leaching recovers F/Na/Al and the remaining carbon or refractory fraction is utilized. Roasting-water-leach and molten-salt-assisted routes fit this architecture (Dong, Jiao, Liu, & Li, 2025; Dong, Jiao, Liu, & Ling, 2025; Lan et al., 2024; Li et al., 2024; Y. Li et al., 2023; Lv et al., 2025; Xu et al., 2023; Xu et al., 2024; K. Yang et al., 2024; Yao et al., 2024; Yao et al., 2020; Zhang et al., 2025; Zhao, Liu, et al., 2021; Zhu et al., 2023; Zhu et al., 2026). The advantage is safer or more reactive material entering wet processing; the disadvantage is the potential volatilization of recoverable fluoride and the energy cost of heating material that may subsequently be in contact with large volumes of water.

Route VI - Direct Co-Processing

Route VI prepares and blends SPL into cement, ceramics, metallurgical furnaces, mold flux, asphalt, or related high-temperature uses (Al Jawi et al., 2020; Birry et al., 2023; Brial et al., 2021; Brial et al., 2022; Camara & Ouellet-Plamondon, 2023; Han et al., 2024; Hou et al., 2021; Khan & Chen, 2025; Sang, Liu, et al., 2022; Tu et al., 2023; Zhang et al., 2026; Zhao, Ma, et al., 2021). Its attractiveness depends strongly on local infrastructure and avoided disposal cost. The decisive acceptance test is not destruction of the SPL identity but a verified pollutant-transfer balance and host-product quality. F volatilization, alkali loading, carbon substitution efficiency, slag/product chemistry, and stack-gas capture must all be demonstrated.

Route VII - Closed-Loop Aluminum-Smelter Recovery

Route VII targets return of SPL-derived materials to the aluminum value chain. In its strongest form, selective leaching or thermal separation produces an F-Al-Na stream that is converted to AlF_3 or cryolite, while the carbon fraction is purified for a qualified cathodic, anodic, ramming-paste, or other carbon application (Dong, Jiao, Liu, & Ling, 2025; Lan et al., 2024; Li et al., 2026; Sommerseth et al., 2025; Yan et al., 2026; Yang et al., 2022; Yang et al., 2023; Zhu et al., 2026). This route has the highest circularity potential because it can displace primary electrolyte and carbon materials, but it also has the tightest purity requirements and requires a plant-wide contaminant-control strategy.

Route VIII - Near-Zero-Waste SPL Refinery Concept

Route VIII combines the most selective elements of the previous architectures and treats SPL as a multi-product feed. Carbon, fluoride, aluminum/sodium salts, refractory minerals, and process water are recovered or recycled separately; only a minimized, characterized residue leaves the system. Recent comprehensive recycling and multi-component separation studies point toward this refinery logic, including simultaneous recovery of carbon and electrolyte, directional conversion of valuable components, and integrated treatment of carbon-rich and refractory fractions (Dong, Jiao, Liu, & Li, 2025; Feng et al., 2025; Li et al., 2026; X. Li et al., 2025; Lu et al., 2020; Tian et al., 2026; Yang et al., 2022; Zhang et al., 2022). The concept is technically attractive but presently suffers from flowsheet complexity and limited long-duration demonstration.

Across the eight architectures, the most important sequencing question is whether hazardous/reactive species should be destroyed before wet treatment or whether soluble value should first be recovered. There is no universal answer. High-CN or highly reactive SPL may justify early detoxification; cleanly segregated first-cut material with recoverable electrolyte may justify selective wet or vacuum separation first. The correct answer emerges from feed characterization, product targets, and constituent-fate analysis.

The quantitative literature is heterogeneous in feed definition, particle size, reagent basis, temperature, and endpoint, so pooled means would be misleading. Table 4 therefore benchmarks selected, directly reported outcomes from representative route studies and retains their evidence level. These values should be interpreted as study-specific demonstrations rather than universally achievable performance.

Table 4. Selected quantitative benchmarks for representative SPL-processing routes

Study/route	Feed and principal process	Evidence level	Directly reported benchmark	Critical interpretation
Yao et al. (2020) Route V	Spent carbon cathode; NaOH-Na_2CO_3 molten-salt roasting + water leaching	L1	F leaching 96.92%; recovered graphitized carbon purity 96.38%.	Strong chemical separation; continuous recycle and utility data remain unproven.
Li et al. (2022) Route V	Spent carbon cathode; low-temperature sulfation roasting + water leaching	L1	F removal 97.82%; recovered carbon purity 90.29 wt%.	High defluorination does not by itself establish product-market qualification.
Lv et al. (2025) Routes II/V	Spent cathode carbon; low-temperature molten-salt roasting + two-stage leaching	L1	Recovered graphite carbon reported at 98.67 wt%.	High purity at the laboratory scale; salt recovery and recycling remain key scale-up questions.
Sommerseth et al. (2025) Route VII	First-cut SPL; 5-19 kg batches, high temperature and reduced pressure	L2	Carbon up to 98.2 wt%; condensate mainly Na_3AlF_6 and NaF.	Rare kilogram-scale evidence; energy, transport, condenser/filter design and product outlet remain decisive.
Dong et al. (2025) Routes V/VII	Overhaul slag; low-temperature H_2SO_4 roasting + water leaching	L1	Cryolite recovery 98.69%; reported product grade 99.34%.	Product-oriented F-Na-Al recovery; impurity and recycle control govern closed-loop feasibility.
Zhu et al. (2026) Route VII	Spent cathode carbon; leaching-mixture calcination route to AlF_3	L1	Total F leaching 99.48%; AlF_3 product purity reported at 93.60 wt%.	Demonstrates targeted fluoride product formation; receiving-smelter specification remains application-specific.

Note. Values are reproduced as reported by the individual studies and are not normalized across feed composition or process boundary. Evidence levels follow Table 1. The table is a benchmark of demonstrated outcomes, not a meta-analysis or commercial guarantee.

Product-Oriented Recovery and Quality Requirements

Recovered Carbon and Graphite

Carbon recovery is only meaningful when the recovered material satisfies an end-use specification. Relevant variables include fixed carbon or graphite content, ash, F, Na, Al, S, particle size, graphitic order, surface chemistry, electrical behavior, calorific value, and, for electrochemical applications, electrochemical performance. The corpus spans conventional graphite concentrate, thermally purified carbon, battery-anode feedstock, regenerated graphite, and prebaked-anode applications (Angelopoulos et al., 2021; Chen et al., 2023; Chen et al., 2024; Li et al., 2026; W. Li et al., 2025; Li et al., 2022; Merchichi et al., 2026; Robshaw et al., 2022; Sundby et al., 2020; Yan et al., 2026; Yang et al., 2020; Zheng et al., 2024; Zhou et al., 2023). These studies collectively show that product quality, not percentage carbon recovery alone, is the correct endpoint.

The achievable market tier depends on the severity of purification. Lower-value uses as fuel, reductant, carburizer, or metallurgical carbon can tolerate more ash and residual salts. Battery or aluminum-anode applications demand deeper removal of fluoride and mineral impurities and may require control of particle morphology or graphitic structure (Chen et al., 2023; Li, Chen, Ouyang, et al., 2025; Robshaw et al., 2022; Yan et al., 2026; Zheng et al., 2024). This creates a classic value-versus-processing-intensity trade-off that must be included in TEA rather than assumed away.

Fluoride, Sodium, Aluminum, and Refractory Products

Fluoride product options include CaF_2, cryolite, AlF_3, and other F-bearing salts. Cryolite preparation, fractional precipitation, targeted CaF_2 separation, and AlF_3 synthesis demonstrate increasingly product-specific recovery strategies (Dong, Jiao, Liu, & Ling, 2025; Dong, Jiao, Liu, & Zhang, 2025; Lan et al., 2024; Ma et al., 2024a; Yang et al., 2023; Zhu et al., 2026). The preferred product depends on liquor composition and local market or smelter demand. Closed-loop use favors electrolyte-compatible products, whereas external markets may accept different purity windows.

Refractory-rich products require a different qualification logic. Treated SPL has been evaluated in cementitious systems, ceramics, asphalt, mold flux, master-alloy production, and other mineral applications (Al Jawi et al., 2020; Birry et al., 2023; Brial et al., 2021; Brial et al., 2022; Camara & Ouellet-Plamondon, 2023; Han et al., 2024; Hou et al., 2021; Khan & Chen, 2025; Tu et al., 2023; Zhang et al., 2026). These applications can capture the Al-Si-Ca mineral value, but are constrained by residual F and Na, leachability, volumetric stability, and host-product performance. The best refractory route may therefore be one that deliberately separates soluble F before mineral utilization.

Table 5 links the main recovered products to the quality parameters and representative quantitative evidence that should be reported before a route is described as recycling rather than treatment. Reported purities are evidence benchmarks, not universal commercial specifications.

Table 5. Recovered products, quality requirements, reported benchmarks, and application constraints

Recovered product	**Key quality parameters**	**Potential application**	**Reported evidence benchmark / major constraint**
Purified carbon/graphite	C/ash, F, Na, Al, S, PSD, graphitic order; application-specific electrochemical or electrical tests.	Metallurgical carbon, ramming paste, electrodes, batteries.	Selected laboratory/bench studies report about 90.29-98.67 wt% C, including 98.2 wt% at kilogram scale; deeper qualification depends on the receiving specification.
AlF_3	F/Al stoichiometry, Na/Ca/Si/Fe impurities, phase quality, moisture.	Aluminum-smelter fluoride make-up.	Zhu et al. (2026) reported 93.60 wt% purity; actual smelter acceptance is specification-specific and may require further purification.
Cryolite	Na:Al:F balance, moisture, foreign ions, phase composition.	Return to Hall-Héroult electrolyte.	Dong, Jiao, Liu, & Ling (2025) reported 98.69% recovery and 99.34% product grade; impurity accumulation in recycle remains critical.
CaF_2 / F mineral product	F grade, solubility/leachability, impurities.	Flux/mineral feed or stable fluoride product.	No single benchmark is transferable across routes; endpoint may represent immobilization rather than closed-loop recycling if no qualified outlet exists.
Na-bearing salts	Na grade, F/Al contamination, purge chemistry.	Internal recycle or external salt use.	No universal product specification; accumulation can determine purge rate, evaporation duty, and long-term water-loop stability.
Refractory/mineral product	F/Na leachability, mineralogy, strength/reactivity and host-product performance.	Cement, ceramics, asphalt, flux, metallurgical feed.	Acceptance is end-use-specific; composition alone is insufficient without environmental and performance testing.

Note. Quantitative values are reported evidence benchmarks, not universal product specifications. Final recycling claims require compliance with the receiving process or customer specification and evidence of actual substitution. Compiled and adapted from carbon-upgrading, fluoride-product, refractory-valorization, and closed-loop studies (Li et al., 2022; Lv et al., 2025; Sommerseth et al., 2025; Dong, Jiao, Liu, & Ling, 2025; Zhu et al., 2026; Birry et al., 2023; Robshaw et al., 2022; Yan et al., 2026).

Cyanide Destruction, Fluoride Management, and Secondary Pollution

Cyanide Destruction and HCN Management

Cyanide destruction must be demonstrated as a chemical transformation, not inferred from a reduction in solid mass. Thermal oxidation, microwave-assisted treatment, calcification, and stabilization/co-processing routes provide evidence for cyanide decomposition under controlled conditions (Z. (Chen et al., 2022; Sang, Liang, et al., 2022; Sang, Liu, et al., 2022; Song et al., 2026; Su et al., 2022). For wet routes, the principal design concern is preventing conditions that liberate HCN and ensuring that washing or leaching does not relocate cyanide to an unmanaged liquor or headspace.

Fluoride Dissolution, Immobilization, Recovery, and Recycling

Four outcomes must be distinguished. Fluoride dissolution transfers F into solution; immobilization converts F into a less mobile solid phase; recovery isolates F in a deliberately separable product; and recycling returns that product to a defined use. Water leaching and acid/alkali leaching demonstrate dissolution (Nie et al., 2020; Shirmahd et al., 2023; Tropenauer et al., 2021; Wang et al., 2023; Xu et al., 2024); calcification and stabilization demonstrate immobilization (Dong, Jiao, Liu, & Zhang, 2025; Sang, Liang, et al., 2022; Song et al., 2026; Zhang et al., 2020); precipitation and cryolite/AlF_3 production demonstrate recovery (Dong, Jiao, Liu, & Ling, 2025; Lan et al., 2024; Ma et al., 2024a; Yang et al., 2023; Zhu et al., 2026); and only those recovered products that meet an end-use specification qualify as recycling. This hierarchy prevents optimistic environmental claims based only on extraction percentage.

Off-Gas, Wastewater, and Secondary Solids

Thermal routes must account for HF or other fluoride-bearing species, particulate matter, NH_3, combustible gases, and condensed electrolyte. Measurements of F-bearing particulate during SPL combustion and studies of fluorine migration show why the gas-cleaning train must be included in both material balance and operating cost (Han et al., 2023; M. Li et al., 2023; Lin et al., 2023; Zhang et al., 2023; Zhu et al., 2023). Low-pressure thermal treatment similarly requires controlled condensation because recovered electrolyte species and possible reactive sodium-bearing condensates cannot be treated as a generic dust stream (Sommerseth et al., 2025).

Hydrometallurgical routes shift the burden toward wastewater. Counter-current washing, liquor recycle, and selective precipitation can reduce fresh water consumption, but Na, F, Al, Ca, sulfate or chloride can accumulate until a purge is required. Closed-circuit concepts and combined leaching/ion-exchange or precipitation systems demonstrate the potential for recycle while also showing that solution chemistry is a flowsheet constraint (Ma et al., 2024a; Robshaw et al., 2020; Yang et al., 2022; Yang et al., 2023). Secondary solids must be characterized for F leachability and residual cyanide before beneficial use.

Table 6 presents a pollutant-transfer matrix. It is designed to prevent a route from appearing environmentally superior merely because a contaminant leaves the original SPL solid.

Table 6. Pollutant-transfer matrix for integrated SPL routes

Process step	Gas-phase transfer	Liquid-phase transfer	Solid/product transfer	Critical control
Crushing/grinding	F-bearing dust; particulate	Minimal unless wet	Fine contaminated solids	Enclosure, dust collection, segregation
Water washing	Possible H_2/CH_4/NH_3 from reactive phases	Soluble F/Na/CN species	Washed carbon/minerals	Gas monitoring; liquor recovery
Acid/alkali leaching	Possible HCN if pH mismanaged	F/Na/Al/Ca/Si + reagents	Purified or partially purified solid	pH control; selective precipitation; recycle
Thermal oxidation/roasting	HF/F particulate, NH_3, combustion gases	Usually indirect via scrubber liquor	Ash, fixed F mineral phases	Off-gas capture and scrubber/sludge balance
Vacuum/low-pressure heat	Volatile electrolyte to condensate	Minimal direct	Purified carbon + condensate	Condenser/filter design; reactive condensate handling
Precipitation	Minimal	Residual salts and mother liquor	CaF_2/cryolite/AlF_3 or sludge	Product purity; mother-liquor recycle/purge
Co-processing	F/alkali/particulate to stack if not retained	Plant-specific	Host product/slag incorporates minerals	Mass balance + product quality + emissions

Note. Conceptual pollutant-transfer matrix adapted from emission-control, leaching, stabilization, thermal-treatment, and whole-process fluoride studies (Han et al., 2023; Lin et al., 2023; Sang, Liang, et al., 2022; Sommerseth et al., 2025; Su et al., 2022; Yang et al., 2022; Zhang et al., 2020).

Constituent-Fate and Process-Integration Analysis

The core performance metric proposed in this review is constituent fate. Each quantitatively important element and, where relevant, hazardous species entering the system should be assigned to a qualified product, a net external aqueous output, an off-gas output, residual solids, or an inventory change. Internal recycle streams remain within the system boundary and therefore cancel from the overall balance. This is more informative than reporting only extraction because it reveals whether the process truly conserves value or merely transfers contamination.

Figure 4 shows the constituent-fate and mass-balance framework used to interpret integrated routes. Arrow width is deliberately non-quantitative; the figure distinguishes external inputs and outputs, internal recycle loops, and inventory change and is therefore a fate-and-boundary map rather than a Sankey diagram.

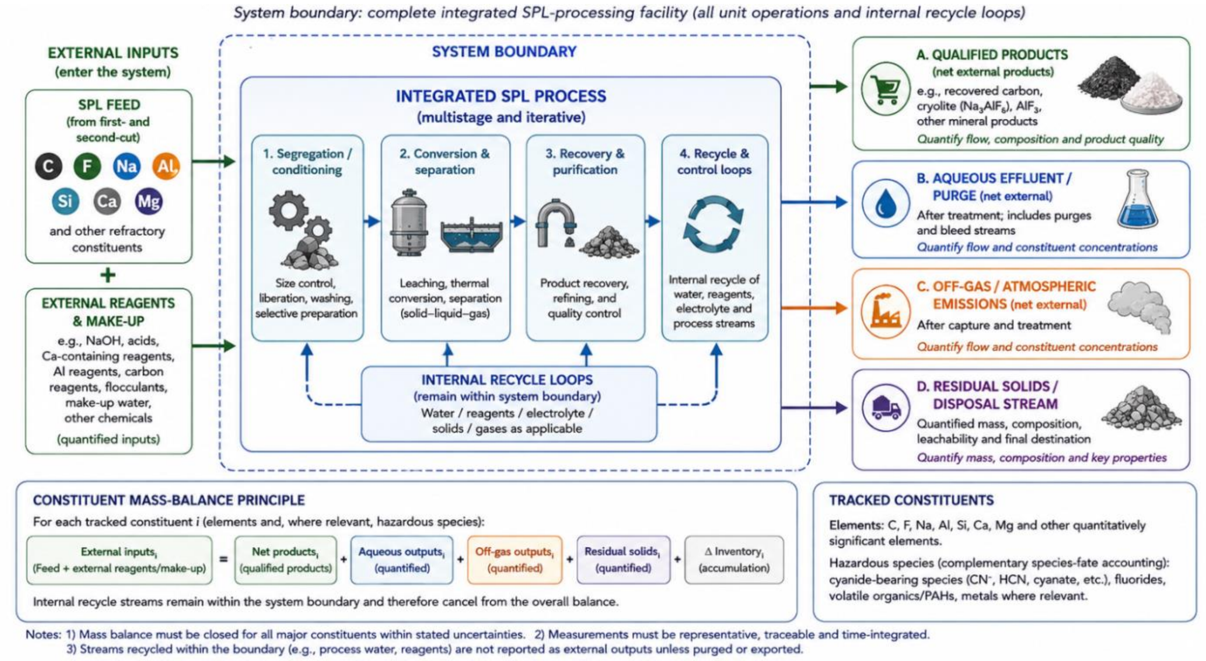

Figure 4. Conceptual constituent-fate and overall mass-balance framework for integrated SPL valorization.

Note. Original conceptual synthesis adapted from material-flow, fluorine-migration, co-treatment, and integrated-recovery studies (Billy et al., 2022; M. Li et al., 2023; Y. Li et al., 2023; Lin et al., 2023; Lu et al., 2020; W. Xie et al., 2020; Zhang et al., 2023; Zhang et al., 2022; Zhao, Ma, et al., 2021).

Carbon Fate

Carbon can be reported to a purified product, oxidized for energy or detoxification, remain in a mineral residue, or be converted into a different carbon material. Gasification and combustion intentionally sacrifice part of the carbon value for energy or hazard control (Deng et al., 2022; Janajreh et al., 2021; Nemmour & Ghenai, 2021; Nemmour et al., 2023), whereas flotation, leaching, vacuum treatment, and hybrid purification aim to preserve carbon as a material (Angelopoulos et al., 2021; Chen et al., 2024; Li et al., 2022; Merchichi et al., 2026; Robshaw et al., 2022; Sommerseth et al., 2025; Sundby et al., 2020; Zhou et al., 2023). The correct route therefore depends on the realistic value of the recovered carbon and the cost of meeting its impurity specification.

Fluorine, Sodium, and Aluminum Fate

Fluorine has the greatest risk of unrecognized transfer because it can move among solution, precipitate, condensate, ash, host product, and off-gas. Fluorine-migration studies in slag and co-treatment systems show that temperature and mineral chemistry can redistribute F rather than eliminate it (M. Li et al., 2023; Y. Li et al., 2023; Zhang et al., 2023; Zhu et al., 2023). Sodium behaves similarly in hydrometallurgical recycling loops, whereas aluminum can be either a valuable component of cryolite/AlF_3 chemistry or an impurity that complicates precipitation. A closed F-Na-Al balance is therefore essential for judging closed-loop claims (Dong, Jiao, Liu, & Ling, 2025; Lan et al., 2024; Lin et al., 2023; Ma et al., 2024a; Yang et al., 2023; Zhu et al., 2026).

Conceptual Constituent Balances

$$M_{i,external\ inputs} = M_{i,net\ products} + M_{i,aqueous\ outputs} + M_{i,off\text{-}gas\ outputs} + M_{i,residual\ solids} + \Delta M_{i,inventory}$$

$$Balance\ closure_i\ (\%) = 100 \times [\Sigma M_{i,external\ outputs} + \Delta M_{i,inventory}] / \Sigma M_{i,external\ inputs}$$

Here, i denotes a tracked constituent. External inputs include SPL feed plus any external reagent or make-up contribution containing i; external outputs include net products, aqueous purge/effluent, off-gas, and residual solids. Internal recycle streams are not counted as external outputs because they remain inside the system boundary; a recycle stream is included only if it is purged or exported. Inventory change in tanks, condensate receivers, dust systems, filter cakes, or stored solids is explicitly reconciled. For hazardous species such as cyanide, species-fate or destruction accounting complements the elemental balance because chemical destruction changes species identity. Pilot and industrial balances should report analytical and sampling uncertainty and a closure criterion, because short campaigns can otherwise conceal Na or F accumulation in recycle inventories.

Process Engineering, Scale-Up, and Techno-Economic Constraints

Solid Handling, Separation, and Materials of Construction

SPL flowsheets are mechanically demanding because abrasive mineral matter, fine carbon, salts, and corrosive leach liquors coexist. Grinding energy and dust control compete with liberation; fine carbon can make filtration difficult; and fluoride/chloride/acid systems constrain materials of construction. Studies that achieve strong chemical performance at small scale rarely provide enough information on slurry rheology, filter area, cake washing, equipment fouling, or corrosion to support direct scale-up (Dzikunu et al., 2023; Merchichi et al., 2026; Sathish et al., 2025; Yang et al., 2022). These are not secondary details: they often determine whether a chemically selective route is operable.

Reagent, Water, and Thermal-Energy Demand

Hydrometallurgical routes incur costs for acid/alkali, neutralization, water, and evaporation or crystallization. Thermal routes incur sensible heat, reaction heat, furnace losses, gas handling, and, for vacuum systems, pressure-maintenance costs. Process-intensified systems can shorten residence time but may exchange bulk energy demand for high-power ultrasound, microwave, or Joule-heating equipment (Huang et al., 2022; Nemmour et al., 2023; Tian et al., 2025; Tian et al., 2024; Xu et al., 2023; Zhu, Xu, Han, Liu, Zhang, Tian, et al., 2022; Zhu, Xu, Han, Liu, Zhang, Yang, et al., 2022). The relevant comparison is therefore energy and reagent consumption per tonne of representative SPL and per tonne of qualified product, not per laboratory batch.

The low-pressure thermal study is instructive because its LCA identified treatment energy, SPL preprocessing, and transport distance as major sustainability variables (Sommerseth et al., 2025). This illustrates why plant location and integration with smelter infrastructure can be as important as reactor efficiency. A process that returns recovered electrolyte or carbon to the same site can earn both transport and substitution benefits that a remote standalone facility cannot.

Product Revenues, Disposal Avoidance, and Scale-Up Evidence

The economic value stack combines avoided hazardous-waste storage/disposal, recovered carbon value, fluoride-product value, potential mineral-product value, and avoided purchase of primary materials. Industrially oriented work such as the low-caustic leaching and liming process and the HiCAL30 solution demonstrates the importance of linking treatment to a stable product or feedstock outlet (Birry & Gauthier, 2024; Shareef et al., 2023). Conversely, high-purity laboratory products do not guarantee profitability if the purification chain is reagent-intensive or the market is too small.

Table 7 summarizes the main process-engineering and techno-economic drivers together with the availability of evidence in the reviewed corpus. The additional evidence-availability column prevents absence of reported data from being interpreted as low cost or low environmental burden.

Table 7. Process-engineering, techno-economic drivers, and evidence availability for integrated SPL routes

Indicator	Dominant hydrometallurgical/thermal drivers	Why it matters for scale-up	Evidence availability in core corpus
Feed preparation	Liberation, PSD, slurry solids/particle size, feeding, heat transfer, dust.	Controls kinetics, handling, throughput, and representativeness.	Moderate
Reagent/utility intensity	Acid, alkali, neutralant, wash water/fuel, electricity, carrier gas, vacuum.	Major OPEX and footprint driver; should be normalized per tonne SPL and qualified product.	Low-Moderate
Separation equipment	Thickening, filtration, washing/cyclones, filters, condensers.	Determines recovery losses, availability, fouling, and operability.	Low
Recycle stability	Na/F/Al/sulfate/chloride accumulation/condensate recycle and gas recirculation.	Defines purge requirement and long-term circuit chemistry.	Low
Materials of construction	Corrosion/erosion in leach circuits / high-temperature and fluoride exposure.	Influences CAPEX, maintenance, reliability, and contamination.	Low
Product qualification	Carbon and precipitate purity, condensate chemistry, ash/mineral quality.	Determines realizable revenue and whether recovery qualifies as recycling.	Moderate
Environmental controls	Effluent treatment and purge/off-gas, particulate, scrubber and condensate control.	Prevents burden shifting among air, water, and solids.	Moderate
Scale evidence	Continuous leach/recycle campaign / large-batch or continuous furnace campaign.	Reduces extrapolation uncertainty and exposes inventory/fouling effects.	Very low
Economics and LCA	Reagents, water, residue disposal/energy, gas handling, maintenance; avoided disposal and material substitution for all routes.	Requires site-specific boundary, energy source, transport, product price, and uncertainty.	Very low

Note. Evidence availability is a qualitative description of reporting density in the 106-study core corpus, not a score of process performance. The recurring weakness is missing normalized utility, recycle, corrosion, and cost data even when chemistry is well demonstrated. Framework adapted from integrated leaching, industrial valorization, low-pressure thermal, gasification, and process-intensification studies (Birry & Gauthier, 2024; Dzikunu et al., 2023; Merchichi et al., 2026; Nemmour et al., 2023; Sathish et al., 2025; Shareef et al., 2023; Sommerseth et al., 2025).

Environmental Performance, Circularity, and Regulatory Constraints

Hazard Elimination Versus Waste Transfer

Environmental performance cannot be inferred from a lower mass of solid waste. Cementitious and other co-processing routes can produce genuine substitution benefits, but they must demonstrate the fate and leachability of fluoride and the effect of alkalis on the host system (Al Jawi et al., 2020; Birry et al., 2023; Brial et al., 2021; Brial et al., 2022; Camara & Ouellet-Plamondon, 2023; Han et al., 2024; Tu et al., 2023). Hydrometallurgical routes can recover fluoride and carbon while increasing water and reagent footprints; thermal routes can eliminate cyanide while increasing energy demand or transferring F to off-gas. Circularity therefore requires multi-media accounting.

Avoided Primary Materials and Circularity Hierarchy

The strongest environmental credit arises when recovered SPL products displace primary materials: purified carbon can replace virgin or conventionally processed carbon in suitable applications; AlF_3 or cryolite can reduce demand for primary fluoride inputs; CaF_2 can displace fluorspar in applications that accept the product; and treated refractory fractions can replace mineral raw materials (Dong, Jiao, Liu, & Ling, 2025; Hou et al., 2021; Lan et al., 2024; Robshaw et al., 2022; Tu et al., 2023; Yan et al., 2026; Zhang et al., 2026; Zhu et al., 2026). The magnitude of this benefit depends on product quality and actual market substitution, not theoretical composition.

Figure 5 expresses the circularity hierarchy used in this review. Each higher level requires the conditions of the level below it plus stronger evidence of material substitution and loop closure.

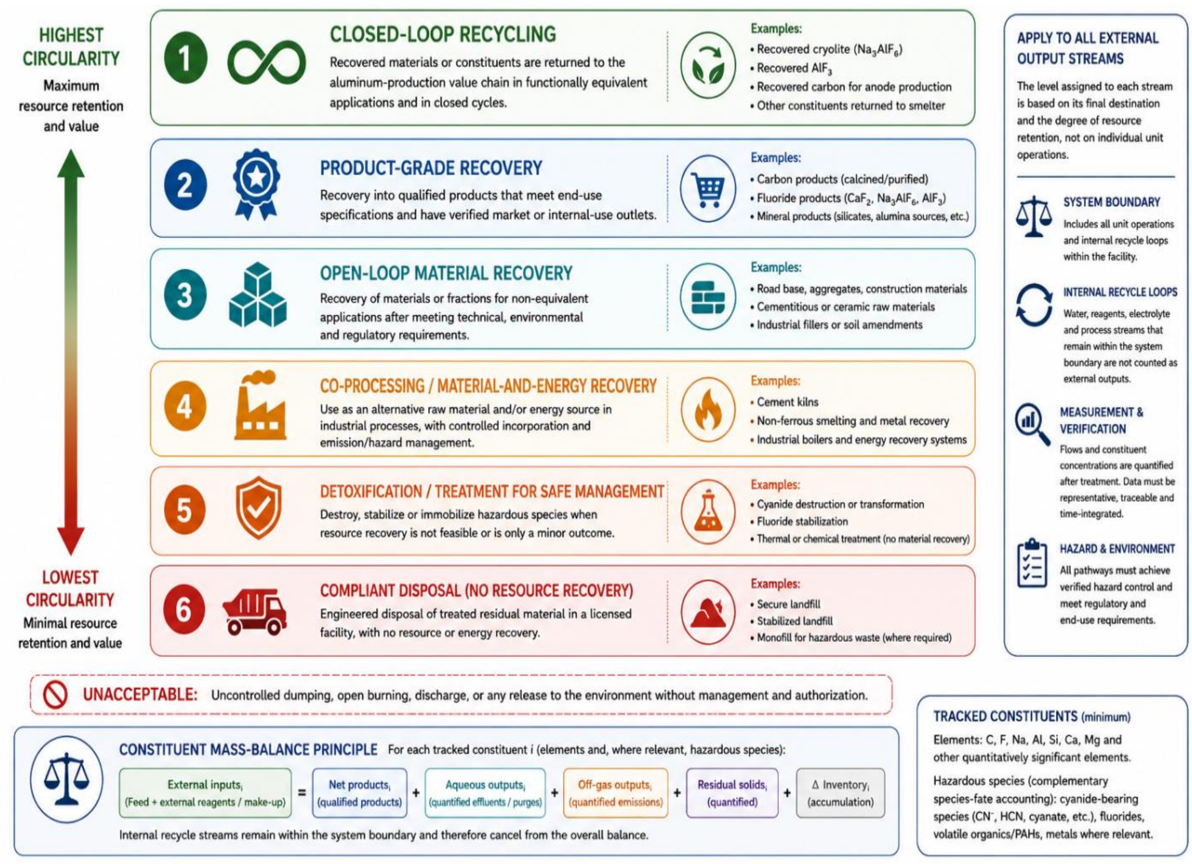

Figure 5. Transition from SPL disposal to closed-loop circular processing.

Note. Original hierarchy developed for this review and adapted from sustainable SPL management, cement valorization, carbon recovery, and closed-loop process studies (Al Jawi et al., 2020; Nunez, 2020; Pereira, 2025b; Shareef et al., 2023; Yang et al., 2022).

Regulatory, Product-Acceptance, and Sustainability Implications

Regulatory status is jurisdiction-specific and should not be generalized from one country to another. In the United States, spent potliners from primary aluminum reduction are classified as hazardous waste K088 and subject to land-disposal treatment requirements; this provides a useful example of the distinction between verified hazard treatment and authorization of a recovered material for a new use (U.S. EPA, 2000). Outside that framework, project development must verify the applicable waste classification, transport and storage requirements, leachability or emission limits, end-of-waste or by-product criteria where relevant, and the receiving industry's product specification.

Across jurisdictions, fluoride and cyanide remain central hazard-control concerns, while circular use adds a separate product-acceptance obligation (Liu, 2020; Nunez, 2020; Ospina &Hassan, 2017; Pereira, 2025b). A material that is legally treated but fails a customer's impurity or performance specification remains a residue-management problem; conversely, a technically high-value product that retains excessive leachable fluoride or uncontrolled cyanide cannot be considered a safe secondary raw material.

System-level environmental accounting is therefore preferable to single-impact claims. Material-flow analysis at smelter scale and LCA-oriented work on SPL treatment show that transportation, energy source, preprocessing, product substitution, and emission-control choices can materially change the apparent benefit of a route (Billy et al., 2022; Lin et al., 2023; Sommerseth et al., 2025). The preferred architecture is likely to be site-specific, with strong advantages for solutions that are physically and logistically integrated with aluminum production.

Critical Comparison, Route Selection, and Research Priorities

No Universal Best Route

The evidence does not support a universal 'best SPL technology.' The optimum route is feed-specific and product-specific rather than technology-specific. Carbon-rich first cut should generally be evaluated first for carbon preservation plus electrolyte removal; fluoride-rich material should prioritize F-Na-Al recovery; refractory-rich second cut should prioritize mineral recovery or controlled co-processing; high-cyanide or highly reactive SPL may require an early hazard-control step; and heterogeneous mixed SPL benefits from stronger front-end segregation.

This conclusion reconciles the apparent diversity of the literature. Physical flotation (Angelopoulos et al., 2021; Chen et al., 2024), successive leaching (Dzikunu et al., 2023; Sathish et al., 2025), vacuum/low-pressure heat (Sommerseth et al., 2025; Sundby et al., 2020; Xin et al., 2022), molten-salt and microwave treatment (Li et al., 2024; Lv et al., 2025; Tian et al., 2024; Xu et al., 2023; Zhu, Xu, Han, Liu, Zhang, Tian, et al., 2022; Zhu, Xu, Han, Liu, Zhang, Yang, et al., 2022), closed-circuit recovery (Yang et al., 2022), and direct co-processing (Al Jawi et al., 2020; Han et al., 2024; Sang, Liu, et al., 2022; Tu et al., 2023) are not competing answers to the same feed-and-product problem. They occupy different positions in a route-selection space defined by composition, hazard, product target, infrastructure, and scale.

Integrated Route Assessment Matrix

Table 8 compares six consolidated route families derived from the eight architectures in Section 6. For compact comparison, Routes I and II are grouped as Hydro; Route III as Thermal; Route IV as Hydro + thermal; Route V as Thermal + hydro; Route VI as Co-processing; and Route VII as Closed-loop. Route VIII is treated as a system-integration superstructure that combines modules from the other families and is therefore not scored as a stand-alone column. Two distinct concepts are reported: process-performance potential and evidence confidence. All performance-oriented rows are directionally positive, so a higher rating always indicates a more favorable attribute. Performance ratings follow the rubric: Very high = the route directly enables the criterion and is repeatedly supported by favorable evidence; High = consistently favorable direct evidence but with scale or boundary limitations; Medium = conditional or mixed performance; Low = weak alignment or an inherent route disadvantage. Evidence confidence is rated High when supported by sustained industrial/demonstration evidence or repeated multi-scale observations, Medium when supported mainly by multiple Level 1-2 studies, and Low when evidence is sparse or primarily conceptual. This separation avoids treating a promising but immature route as equivalent to a mature one.

Table 8. Critical decision matrix for consolidated SPL-processing route families: favorable performance potential and evidence confidence

Criterion	Hydro	Thermal	Hydro + thermal	Thermal + hydro	Co-processing	Closed-loop
CN destruction	Medium	High	High	High	High*	High
F recovery	High	Low-Medium	High	High	Low	Very high
Carbon recovery	High	Low-Medium	Very high	High	Low	Very high
Na management	Medium	Medium	High	High	Low-Medium	Very high
Energy efficiency / low energy demand	High	Low	Low	Low	Medium	Low-Medium
Low chemical demand	Low	High	Low-Medium	Medium	High	Low-Medium
Low water demand	Low	High	Low-Medium	Medium	High	Medium
Low secondary-residue risk	Medium	Medium	High	High	Medium	High
Product-value potential	High	Medium	Very high	High	Medium	Very high
Ease of industrial implementation	Medium	Medium	Low	Low	High	Low
Highest explicit evidence	L1	L1	L1	L1	L4-L5	L2
Overall evidence confidence	Medium	Medium	Medium	Medium	Medium-High	Low-Medium

Note. *CN destruction in co-processing must be demonstrated under actual host-process conditions. All performance-oriented criteria are expressed so that higher ratings are more favorable. Hydro consolidates Routes I-II; Thermal = Route III; Hydro + thermal = Route IV; Thermal + hydro = Route V; Co-processing = Route VI; Closed-loop = Route VII. Route VIII is a multi-product system-integration superstructure and is not scored independently. The strongest explicit evidence aligns with the architecture coding in Figure 3; no Level 3 integrated SPL pilot campaign was identified. Industrial analogues may inform interpretation but do not raise the SPL-specific evidence level. Qualitative synthesis adapted from the full evidence corpus (Chen, Li, Bu, Chehreh Chelgani, et al., 2022; Dong et al., 2023; Khan et al., 2026; Ma et al., 2024b; Pereira, 2025a; Pereira, 2025b; Sommerseth et al., 2025; Yang et al., 2022; Zhou et al., 2024).

Major Knowledge Gaps

- Representative pilot-scale datasets remain sparse. Many studies use carefully selected spent cathode carbon rather than the variable material that an industrial plant must receive.
- Full constituent balances are uncommon. Fluoride extraction is reported far more often than the complete F fate across product, recycle, residue, wastewater, and off-gas streams. Sodium balance and recycle-loop accumulation are underreported even though Na can determine purge rate, evaporation duty, precipitation selectivity, and long-term water-circuit stability.
- Gas-emission inventories are incomplete for several thermal routes, especially where volatile or particulate fluorides and reactive condensates can form.
- Product-quality validation often stops at composition. More end-use testing is needed for recovered carbon, smelter-grade fluoride products, and mineral products.
- CAPEX/OPEX data are limited and are rarely linked to uncertainty in feed composition, product price, disposal avoidance, energy source, or scale.
- Coupled TEA-LCA studies are scarce, and long-duration demonstrations of genuine closed-loop reuse remain exceptional.

Research Roadmap

Because 85 of the 106 core records are conservatively classified as Level 1 and no Level 3 integrated pilot campaign was identified under the present coding, the most useful next experiments are not additional one-factor optimization studies on chemistries already demonstrated. Priority should shift to integrated campaigns that connect representative feed characterization, selective preparation, hazard destruction, constituent recovery, product qualification, water/reagent recycle, off-gas and wastewater control, and sustained pilot validation. Emerging studies provide strong individual modules, but the field now needs those modules assembled into balanced, controllable circuits with explicit operating inventories and failure modes (Li et al., 2026; X. Li et al., 2025; Merchichi et al., 2026; Tian et al., 2026; Yan et al., 2026; Zhu et al., 2026).

Figure 6 converts the critical findings into a route-selection decision tree. The tree deliberately begins with feed identity and hazard profile rather than with a preferred technology.

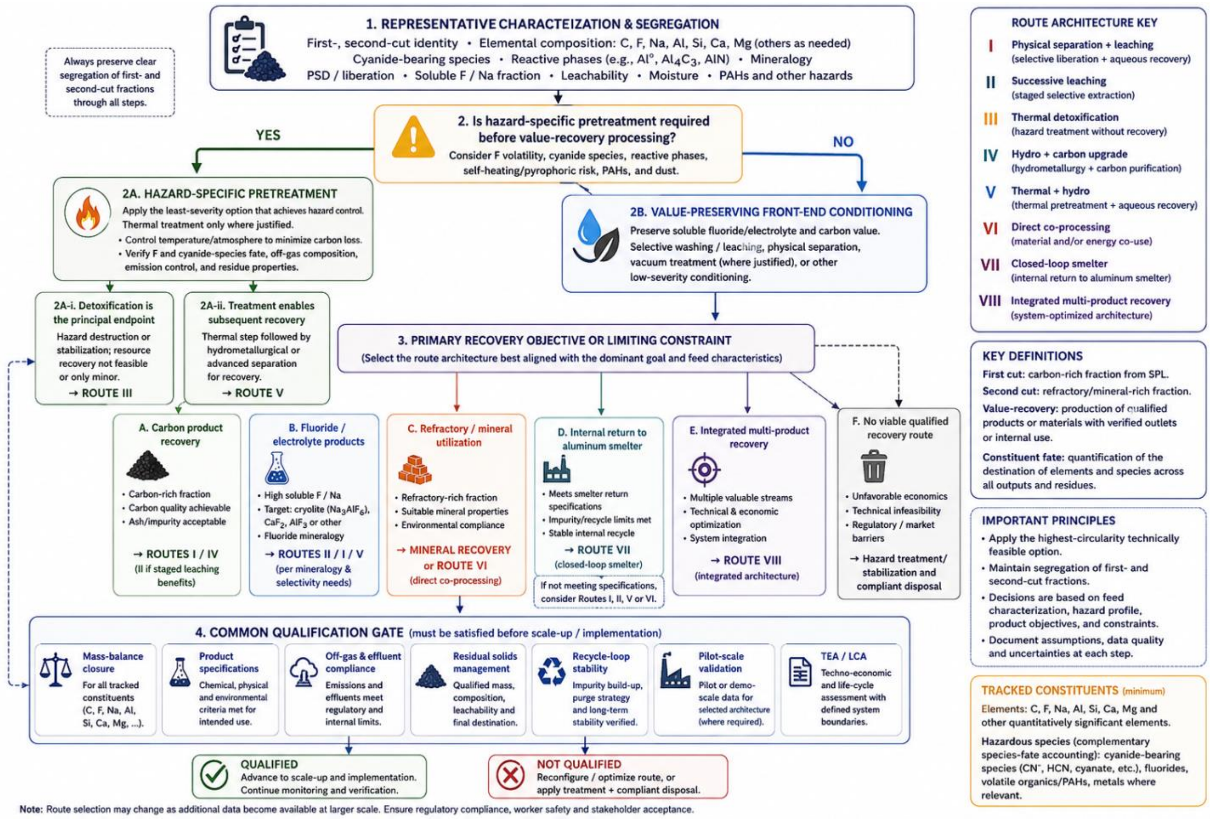

Figure 6. Decision tree for selecting an integrated SPL treatment and valorization route.

Note. Original decision framework developed for this review and adapted from the complete SPL treatment and resource-recovery corpus (Chen, Li, Bu, Chehreh Chelgani, et al., 2022; Dong et al., 2023; Khan et al., 2026; Ma et al., 2024b; Pereira, 2025a; Pereira, 2025b; Zhou et al., 2024).

A credible pilot program should therefore report at least: representative feed mineralogy and variability; all major input and output masses; C/F/Na/Al balances; gas and wastewater composition; reagent and energy use; recycle chemistry over multiple residence-time equivalents; product-quality testing; and a preliminary TEA/LCA. These data would allow the field to move from isolated technical feasibility toward industrial route selection.

Conclusions

Spent pot lining should be treated as a heterogeneous process feed rather than as a uniform hazardous waste. Separating carbon-rich first cut from refractory-rich second cut before intensive processing preserves downstream selectivity and avoids unnecessary reagent or energy consumption. The preferred treatment sequence is therefore determined first by feed composition and hazard profile, and only then by the choice of unit operations.

The integrated routes with the strongest resource-recovery potential are those that preserve carbon value while deliberately recovering the F-Na-Al inventory and controlling cyanide-bearing and water-reactive species. Hydrometallurgical routes offer strong selectivity and product-oriented fluoride recovery, but they must solve water, reagent, and salt-recycle burdens. Thermal routes are powerful for hazard destruction and mineral transformation, but they must account for energy use, fluoride partitioning, condensates, and off-gas. Hybrid routes are most compelling when each step solves a defined limitation of the other rather than merely adding complexity.

The ultimate destination of each element is the decisive test of circularity. Carbon, fluorine, sodium, and aluminum should be tracked to qualified products, internal recycle, controlled emissions/effluents, or characterized final residues. A high extraction percentage without that fate map is insufficient evidence of successful valorization.

The architecture with the greatest long-term closed-loop potential combines selective dismantling, feed-specific hazard control, targeted dissolution or thermal separation, recovery of smelter-compatible fluoride products, qualification of the carbon fraction, recycle of process water and reagents, and minimization of refractory residues. Achieving this outcome will require representative pilot-scale campaigns, complete constituent balances, stable recycle-loop operation, end-use product testing, and integrated techno-economic and life-cycle assessment.

Declarations

Funding

No external funding was received for this review.

Conflicts of Interest

The author declares no conflict of interest.

Data Availability

No new experimental datasets were generated. The study-level evidence coding, evidence scale, primary route assignment, and evidence theme for the fixed 106-publication core corpus are provided in Supplementary Table S1; all underlying source information is derived from the published literature listed in the References section.

Author Contributions

Conceptualization, methodology, literature analysis, critical synthesis, writing, and review: author.

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