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Recovery and Recycling of Spent Ni–Mo–V Catalysts: Technologies, Environmental Drivers and Circular Economy Perspectives

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ABSTRACT **Original Research Article**

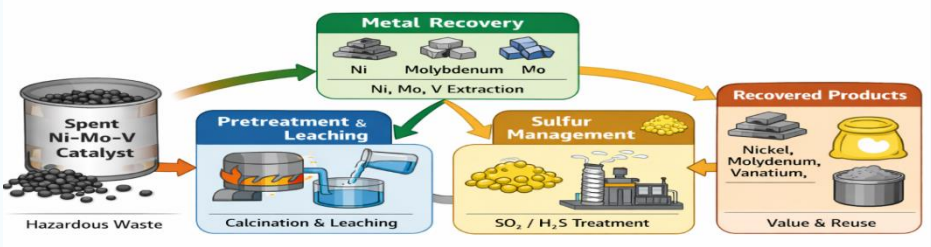
Spent nickel–molybdenum–vanadium (Ni–Mo–V) sulfide catalysts are widely used in hydrotreating, hydrodesulfurization and residue-upgrading units across the global refining industry, producing large volumes of solid residues that are both hazardous waste and valuable secondary resources rich in strategic metals. The presence of sulfides, coke deposits, and toxic trace elements poses significant environmental and health risks if improperly managed, while conventional disposal or partial treatment often results in the irreversible loss of nickel, molybdenum, vanadium, and sulfur. This critical review examines current and emerging technologies for the recovery and recycling of spent Ni–Mo–V catalysts, covering pyrometallurgical, hydrometallurgical and hybrid processing routes, with particular emphasis on selective metal extraction and process integration. Special attention is given to sulfur management, which is often treated as an emission-control issue rather than as a recoverable resource, highlighting opportunities for elemental sulfur recovery through controlled oxidation, Claus-based systems and wet oxidation processes. Key technological limitations related to selectivity, emission control, process scalability and economic viability are identified, and the role of spent Ni–Mo–V catalyst recycling within circular-economy frameworks is critically assessed, emphasizing regulatory drivers and future research directions required to enable fully integrated and sustainable resource recovery strategies.

Keywords: Spent catalysts, Ni–Mo–V, Catalyst recycling, Metal recovery, Circular economy.

Highlights

1. Spent Ni–Mo–V catalysts represent both hazardous waste and a strategic secondary source of critical metals.
2. Hydrometallurgical and hybrid routes enable selective recovery of Ni, Mo and V with lower environmental impact.
3. Sulfur management remains a key bottleneck, often treated as an emission issue rather than a recoverable resource.
4. Integrated metal–sulfur recovery flowsheets improve resource efficiency and circularity.
5. Regulatory pressure and ESG drivers are accelerating innovation in catalyst recycling technologies.

Graphical abstract



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Introduction

Sulfided Ni–Mo–V catalysts are central to modern refining. They enable deep hydrotreating in hydrodesulfurization (HDS), hydrodenitrogenation (HDN), and hydrodemetallization (HDM) units, where performance requirements have tightened as crude slats have grown heavier and product specifications more stringent (Ahn et al., 2023; Alwan et al., 2024). In this context, catalyst management is no longer routine maintenance. It is a strategic lever that links operational reliability, compliance, and material efficiency (Park et al., 2025).

The scale of spent catalyst generation is both significant and persistent. These materials concentrate valuable metals, mainly Ni, Mo, and V, while also containing sulfur species, coke, and refinery-derived contaminants that complicate handling and processing (Puello-Polo et al., 2020; Park et al., 2025). As a result, spent hydroprocessing catalysts are commonly regulated as hazardous waste streams, yet they also serve as a secondary "urban ore" due to their metal grades and supply stability (Zhang et al., 2020; Ying et al., 2025). This dual identity creates tension. Disposal pathways aim to reduce risk, whereas recovery pathways aim to capture value and promote circularity.

Economic and environmental arguments now converge on the need for recovery. On the one hand, primary extraction of Ni, Mo, and V entails large environmental footprints and supply risks. On the other hand, recovery from spent catalysts can reduce upstream burdens, provided that processing routes control emissions, manage residues, and deliver marketable products (Amato et al., 2025; Aromaa-Stubb et al., 2025). Recent life-cycle studies reinforce that the sustainability case depends on the full chain, including transport, pretreatment, reagent use, and by-product management, rather than on metal yield alone (Baritto et al., 2025; Aromaa-Stubb et al., 2025). This means that "recycling" is not necessarily more environmentally benign. It must be demonstrated with transparent boundaries and data.

Circular-economy framing in the refining sector is maturing. Approaches now extend beyond "recover metals" to include remanufacturing, catalyst life extension, and system-level integration of waste and by-product loops (Ippolito et al., 2023). For Ni–Mo–V catalysts, these developments raise practical questions. When is remanufacturing technically credible? When is Full Metal Recovery preferable? Which routes best balance selectivity, reagent intensity, and residue stabilization? These questions are increasingly shaped by ESG reporting and regulatory scrutiny, where traceability and risk management matter as much as recovery rates (Ippolito et al., 2023; Amato et al., 2025).

This critical review evaluates recovery and recycling pathways for spent Ni–Mo–V hydroprocessing catalysts, with emphasis on: (i) the technical constraints imposed by catalyst chemistry and deactivation, (ii) the comparative performance of pretreatment, pyro- and hydrometallurgical routes, and (iii) sustainability evidence, including life-cycle and GHG perspectives that distinguish promising routes from superficially "green" options (Amato et al., 2025; Aromaa-Stubb et al., 2025; Baritto et al., 2025; Ying et al., 2025). We also discuss where circular strategies, such as remanufacturing, fit and where they fail under realistic refinery conditions (Ahn et al., 2023; Park et al., 2025).

The next section describes the review methodology used to identify, screen, and synthesize the literature, and to structure the critical comparison across technologies and sustainability claims.

Methodology

This review follows a structured, transparent protocol aligned with the PRISMA 2020 guidelines for systematic reviews. The methodology was designed to ensure reproducibility, minimize selection bias, and support a critical rather than purely descriptive synthesis of the literature (Page et al., 2021).

Protocol, Search Strategy, Databases, and Eligibility Criteria

The review protocol was defined before literature collection, in line with PRISMA 2020 recommendations (Page et al., 2021). Searches were conducted in major scientific databases, including Web of Science, Scopus, and ScienceDirect, and complemented by targeted searches in ACS Publications, Elsevier, and SpringerLink to ensure coverage of journals in catalysis, metallurgy, and sustainability.

Search strings combined keywords related to spent catalysts, Ni–Mo–V, metal recovery, recycling, hydrometallurgy, pyrometallurgy, sulfur management, and the circular economy. Boolean operators and database-specific filters were used to refine results. Only peer-reviewed journal articles, authoritative reviews, doctoral theses, and selected conference proceedings were considered.

Eligibility criteria were defined as follows:

(i) explicit focus on spent hydroprocessing or hydrogenation catalysts containing Ni, Mo, and/or V;
(ii) discussion of recovery, recycling, remanufacturing, or reuse pathways;
(iii) presentation of experimental, pilot-scale, industrial, or life-cycle data; and
(iv) publication in English.

Studies focused solely on catalyst synthesis without end-of-life considerations or on unrelated catalyst systems were excluded. No strict temporal cutoff was applied, but emphasis was placed on literature published in the past decade to reflect current regulatory, technological, and sustainability contexts.

Figure 1 summarizes the literature selection workflow, from initial database searches to the final set of studies included in the qualitative synthesis.

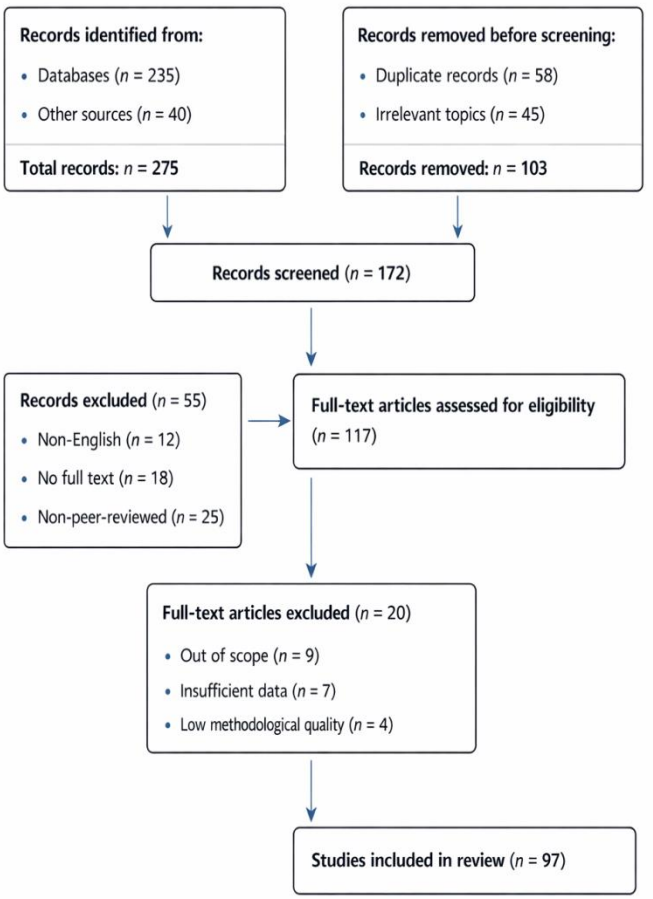

Figure 1. PRISMA 2020 flow diagram illustrating the literature identification, screening, eligibility assessment, and inclusion process used in this review. Adapted from Page et al. (2021).

The PRISMA-based workflow highlights the progressive reduction of records through duplicate removal, abstract screening, and full-text assessment. This structured filtering retained only studies directly relevant to the recovery and recycling of spent Ni–Mo–V catalysts for analysis, while maintaining transparency in exclusion decisions.

Screening, Quality Appraisal, Data Extraction, and Synthesis Approach

Following initial screening, full-text articles were assessed for technical relevance and methodological rigor. Rather than applying a single quantitative scoring system, the quality appraisal focused on the clarity of the experimental design, completeness of mass balance and recovery data, treatment of residues, and consistency between stated objectives and conclusions. This approach aligns with established practices in critical reviews of spent catalyst recycling and secondary metal recovery (Le & Lee, 2021; Liang et al., 2022).

Data extraction focused on process conditions, recovery efficiencies, reagent consumption, residue characteristics, and scalability indicators. When available, techno-economic and life-cycle information was also captured to support cross-comparison. To structure the synthesis, studies were grouped into thematic categories—pretreatment and regeneration, pyrometallurgical enrichment, hydrometallurgical recovery, sulfur management, and circular strategies—following frameworks proposed in recent comprehensive reviews (Pathak et al., 2021; Ying et al., 2025).

This thematic clustering enabled a comparative analysis that goes beyond reporting individual results. It identified recurring bottlenecks, trade-offs between selectivity and process intensity, and gaps between laboratory-scale demonstrations and industrial feasibility. The synthesis, therefore, integrates technical performance with sustainability claims, laying the groundwork for the critical assessment presented in the subsequent sections.

Characteristics of Spent Ni–Mo–V Catalysts

This section examines the origin, composition, and deactivation characteristics of spent Ni–Mo–V hydroprocessing catalysts. The objective is to clarify how formulation, operating severity, and aging mechanisms constrain downstream recovery and regeneration options. Understanding these constraints is essential for judging which recycling strategies are technically credible and which are likely to fail at scale.

Catalyst Types, Formulation, and Operating Context (HDS/HDT/Resid)

Ni–Mo–V catalysts are widely used in hydrodesulfurization (HDS), hydrodenitrogenation (HDN), and residue hydrotreating units operating at high hydrogen partial pressures and elevated temperatures. Typical formulations feature Ni and Mo as active metals, often promoted or contaminated with vanadium during service, and are dispersed on γ-Al_2O_3 or mixed SiO_2–Al_2O_3 supports (Ahn et al., 2023; Alwan et al., 2024). In residue processing, vanadium is not merely a poison but becomes an integral component of the spent catalyst matrix due to continuous deposition from heavy feeds (Puello-Polo et al., 2020).

Operational severity governs both catalyst lifetime and spent catalyst quality. Higher metal loadings and harsher conditions enhance sulfur and nitrogen removal but accelerate deactivation and increase the accumulation of refractory species (Park et al., 2025). This creates a trade-off: catalysts removed from service may contain higher concentrations of recoverable metals, yet they also pose greater challenges for

regeneration and recycling. From a recovery perspective, spent catalysts from resid units are therefore chemically richer but structurally more complex than those from lighter-feed applications.

Table 1 summarizes representative compositions of Ni–Mo–V catalysts reported before and after service, highlighting vanadium enrichment and the persistence of alumina-based supports.

Table 1. Typical composition ranges of fresh and spent Ni–Mo–V hydroprocessing catalysts, including active metals and support materials. Adapted from Ahn et al. (2023), Puello-Polo et al. (2020), Park et al. (2025), Shi et al. (2023).

Component	Fresh catalyst (typical range, wt%)	Spent catalyst (typical range, wt%)	Notes on transformation during service
Ni	1.5–5.0	1.0–4.5	Present mainly as NiO (fresh) and NiS/Ni_3S_2 after sulfidation; partial migration and sintering may occur
Mo	8–15	6–14	Initially as MoO_3; converted to layered MoS_2 under HDS conditions; partial loss via volatilization or entrainment possible
V	<0.5 (trace)	2–15	Accumulates from feedstock (resid feeds); forms VS_x and mixed Ni–V sulfides, strongly associated with deactivation
Co (optional promoter)	0–3	0–3	Remains largely stable; acts as promoter in CoMo systems
Al_2O_3 support	70–85	60–80	γ-Al_2O_3 dominant; structural degradation and surface area loss after aging
SiO_2 (or SiO_2–Al_2O_3)	0–10	0–10	Used to tailor acidity; largely unchanged chemically
Sulfur (total)	<1	5–15	Incorporated during sulfidation; present as metal sulfides and surface sulfur species
Carbon (coke)	~0	5–25	Deposited during operation; pore blocking and diffusion limitation
Fe (contaminant)	<0.5	1–5	Originates from corrosion and feed impurities
As, Pb, Hg (trace metals)	<0.05	<1 (combined)	Strong poisons; accumulate preferentially on active sites

The data show that spent catalysts cannot be treated as uniform materials. Metal ratios, sulfur content, and support chemistry vary widely with feedstock and unit operation, directly affecting the selectivity and reagent demand of recovery processes.

Coke and Sulfur Deposits, Aging, and Regeneration Behavior

Deactivation of Ni–Mo–V catalysts is dominated by coke formation and sulfur accumulation. Coke deposits range from soft, hydrogen-rich species to highly condensed aromatic structures that block pores and encapsulate active sites (Hart, 2021; Shakor et al., 2024). Solid-state NMR and microstructural analyses indicate that coke morphology depends on operating conditions and regeneration history, with wet and dry hydrotreating producing distinct carbon structures (Ghaloum & Ok, 2025).

Sulfur remains largely bound as metal sulfides, such as MoS_2 and NiS_x, but partial oxidation can occur during shutdowns or regeneration attempts, forming mixed sulfide–oxide phases (Abdulla et al., 2025). These transformations complicate recycling. Oxidative regeneration efficiently removes coke but may also promote sintering or phase changes that reduce metal accessibility (Rianto et al., 2022). Non-thermal plasma treatments have been proposed to target refractory coke species, yet their scalability and energy efficiency remain uncertain (Srour et al., 2021).

Figure 2 illustrates the primary deactivation pathways that govern catalyst aging during long-term operation.

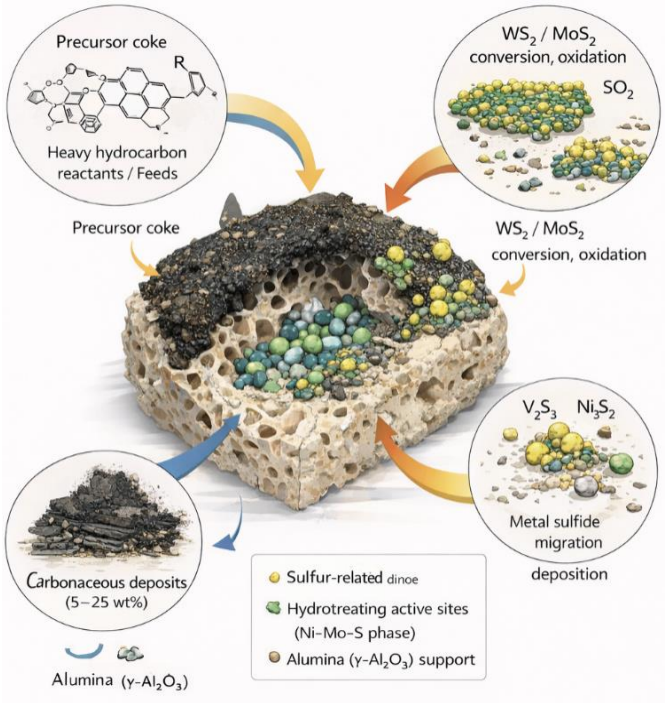

Figure 2. Schematic representation of coke deposition and sulfur-related deactivation mechanisms in Ni–Mo–V hydroprocessing catalysts. Adapted from Hart (2021) and Shakor et al. (2024).

The coexistence of multiple coke types and sulfur species explains why single-step regeneration or leaching often fails. Effective recycling strategies must account for both the chemical diversity of deposits and their spatial distribution within catalyst pellets.

Table 2. Main deactivation mechanisms and regeneration implications. Adapted from Abdulla et al. (2025), Shakor et al. (2024), Hart (2021).

Deactivation factor	Origin	Impact on recovery
Coke deposition	Heavy feeds	Diffusion limitation
Sulfur overload	High-S feeds	SO_2/H_2S emissions
Metal poisons (As, Pb)	Feed contamination	Residue toxicity
Structural collapse	Thermal aging	Lower leachability

Adhered Oils, Contaminants, and Their Implications

In addition to coke and sulfur, spent catalysts retain significant amounts of adsorbed hydrocarbons and trace contaminants. Residual oils can remain trapped in pores or on external surfaces, contributing to mass loss during pretreatment and generating secondary waste streams if not properly managed (Feng et al., 2022; Gao et al., 2022). Recent studies indicate that oil recovery before crushing or leaching can improve downstream metal recovery while reducing solvent and reagent consumption (Hong et al., 2025).

Trace metals such as As, Pb, Hg, and Fe further complicate processing. These elements may poison catalysts during service and subsequently interfere with hydrometallurgical selectivity or solvent-extraction circuits. Their presence reinforces the classification of spent catalysts as hazardous waste and underscores the need for controlled pretreatment prior to metal recovery.

Overall, spent Ni–Mo–V catalysts are chemically heterogeneous materials shaped by formulation, feedstock, and operating history. This heterogeneity explains why recycling routes that perform well in the laboratory often struggle when applied to real-world industrial waste streams. The following section builds on these characteristics to examine pretreatment and conditioning steps, which are critical for stabilizing the material and enabling selective recovery pathways.

Pretreatment and Conditioning Before Metal Recovery

Pretreatment and conditioning are the most critical stages in any recycling route for spent Ni–Mo–V catalysts. Errors at this stage propagate downstream, resulting in poor metal selectivity, excessive reagent consumption, or uncontrolled sulfur emissions. This section critically assesses the main pretreatment strategies and their technical limits, with emphasis on oxidation, coke removal, sulfur control, and physical conditioning.

Oxidative Regeneration and Decoking (Thermal and Chemical Routes)

Oxidative regeneration is widely used to remove coke and restore porosity before metal recovery. Controlled air or oxygen treatments convert carbonaceous deposits to CO_2, while sulfides are partially oxidized to SO_2 or sulfate species (Abdulla et al., 2025). Reaction kinetics depend strongly on coke structure, oxygen availability, and temperature ramp rates (Shakor et al., 2024).

Thermal calcination remains the most mature option, but it is not chemically neutral. High temperatures promote sintering of alumina supports and phase transformations of Mo and Ni species, thereby reducing subsequent leachability (Rianto et al., 2022). Moreover, uncontrolled oxidation can create local hot spots and an irreversible loss of metal dispersion. These effects explain why oxidative pretreatment must be designed as a conditioning step rather than a simple cleaning operation.

Non-thermal plasma oxidation has emerged as an alternative for targeting refractory coke without excessive bulk heating. Plasma-generated radicals can fragment condensed carbon structures that are resistant to conventional calcination (Srour et al., 2021). However, plasma systems pose new challenges in energy efficiency, reactor design, and scale-up. At present, they remain better suited for selective regeneration than for large-scale catalyst recycling.

Figure 3 contrasts the dominant reaction pathways involved in conventional thermal oxidation and plasma-assisted decoking.

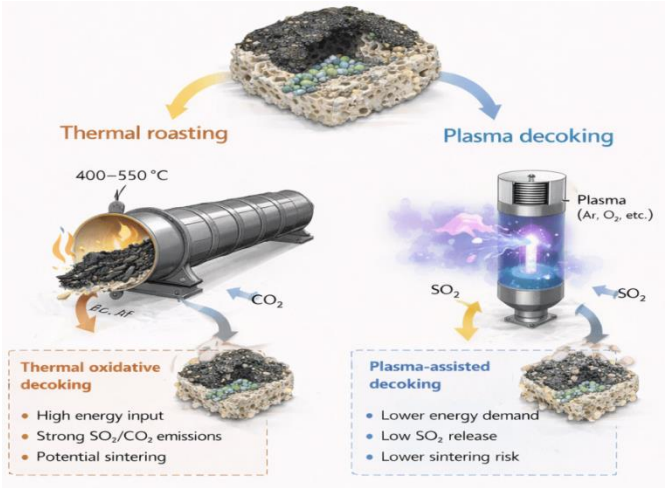

Figure 3. Comparison of oxidative decoking pathways for spent Ni–Mo–V catalysts, highlighting thermal and plasma-assisted mechanisms. Adapted from Abdulla et al. (2025) and Srour et al. (2021).

The figure shows that although plasma routes reduce thermal stress, they do not eliminate sulfur oxidation. As a result, off-gas management remains a central issue regardless of the oxidation strategy used.

Plasma and Other Advanced Pretreatment Routes

Beyond plasma oxidation, several advanced pretreatments have been proposed to improve downstream metal recovery. These include chemical oxidation with mild oxidants, solvent-assisted decoking, and hybrid thermal–chemical schemes. Their common objective is to weaken the interaction between deposits and the catalyst matrix without damaging the support.

Plasma-assisted approaches have been extended to sulfur-containing systems to convert sulfur species under controlled atmospheres (Alqahtani, 2021; Srour et al., 2021). Although promising at the laboratory scale, these methods still raise unresolved questions about reactor durability and integration with conventional hydrometallurgical circuits. In practice, advanced pretreatments are best viewed as complementary tools rather than universal solutions.

Sulfur Behavior under Specific Conditions and Control Strategies

Sulfur behavior during pretreatment determines both environmental performance and process viability. Under oxidative conditions, sulfur is released primarily as SO_2, whereas reducing or hydrothermal environments may favor H_2S formation or redistribution within the solid matrix (Chang, 2024). These transformations complicate sulfur management and impose stringent requirements on gas-handling systems.

Catalytic oxidation of sulfur-containing species has been proposed to mitigate emissions and recover sulfur in controlled forms (Nevanperä, 2021). However, directly coupling these systems to catalyst pretreatment units remains technically complex. The choice of pretreatment atmosphere, therefore, reflects a compromise among effective coke removal, sulfur stabilization, and regulatory compliance.

Comminution and Classification

Mechanical conditioning—such as crushing, milling, and classifying—is required after thermal or chemical pretreatment. Comminution increases surface area and leaching but can release fine particles enriched in sulfur or toxic metals. If unmanaged, these fines can increase reagent use and sludge production.

From a critical standpoint, excessive grinding is rarely justified. Selective size reduction, combined with screening, is often sufficient to expose active phases while preserving downstream operability. Mechanical conditioning should therefore be tailored to the chosen recovery route rather than applied as a default step.

In summary, pretreatment is not a mere formality but a decisive design choice that governs sulfur release, metal accessibility, and overall process robustness. These considerations naturally lead to the next section, which examines the thermodynamic and kinetic foundations of metal-sulfur transformations during recovery operations.

Pretreatment and conditioning are crucial for recycling spent Ni–Mo–V catalysts, as they affect metal accessibility, sulfur behavior, selectivity, and environmental impact. Although often seen as auxiliary, poor conditioning can cause inefficiency, emissions, and metal loss. Table 3 reviews key strategies, their goals, and limitations.

Table 3. Pretreatment strategies and their functional objectives. Adapted from Srour et al. (2021), Rianto et al. (2022), Alqahtani (2021).

Pretreatment	Main objective	Risks/limitations
Oxidative roasting	Sulfide → oxide	SO_2 emissions
Decoking	Restore porosity	Sintering
Plasma	Selective removal	Cost, scale
Grinding/classification	Surface exposure	Dust/toxicity

Thermodynamic and Kinetic Background

This section outlines the thermodynamic limits and kinetic constraints that govern the transformation and recovery of Ni–Mo–V species from spent catalysts. The objective is to define realistic operating windows and explain why several routes fail at scale, even when thermodynamically feasible.

Stability of Ni–Mo–V Sulfides and Oxides

Spent hydroprocessing catalysts contain metals predominantly as sulfides (MoS_2, NiS/Ni_3S_2, and VS_x), which may partially oxidize during operation or pretreatment. Thermodynamic predominance diagrams in the **S–O–M** system indicate that small variations in oxygen partial pressure can shift equilibria from sulfides to oxides or sulfates, particularly for vanadium (Zhang et al., 2020; Ying et al., 2025).

pH and redox potential strongly control phase stability during hydrometallurgical processing. Under acidic and oxidizing conditions, Mo and V tend to form soluble oxyanions, while Ni remains as divalent cations or sparingly soluble oxides (Le & Lee, 2021; Liang et al., 2022). This thermodynamic divergence underpins most selective leaching strategies but also explains their sensitivity to uncontrolled oxidation during pretreatment.

Vanadium has a narrow stability window because of its multiple oxidation states. Its tendency to reprecipitate as mixed oxides or salts complicates leaching and solvent extraction if redox conditions are not controlled (Kurniawan

et al., 2024). Therefore, sulfur removal and oxidation control are crucial for downstream separation.

Figure 4 illustrates the contrasting stability domains of Ni, Mo, and V species relevant to spent-catalyst processing

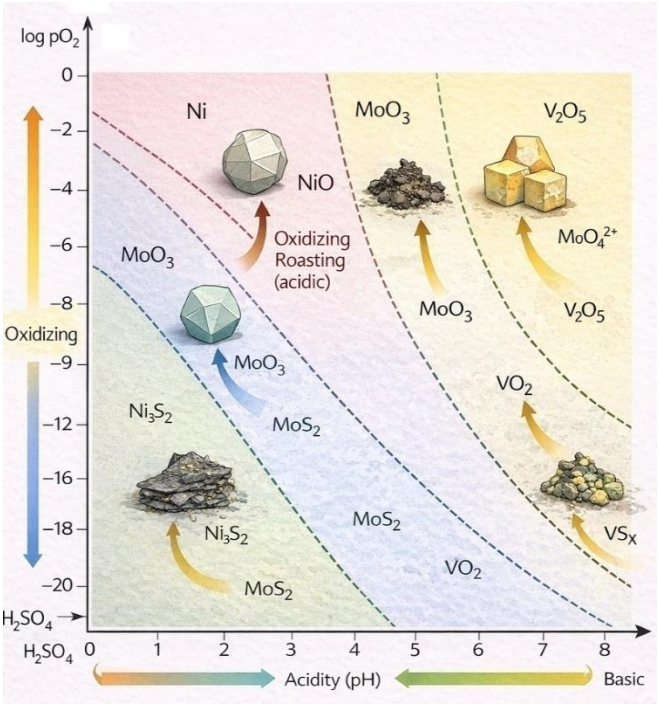

Figure 4. Schematic predominance fields of Ni, Mo, and V sulfide–oxide systems as a function of oxygen potential and acidity. Adapted from Zhang et al. (2020) and Liang et al. (2022).

The diagram explains why single-step treatments rarely achieve full selectivity. Conditions favorable for Mo and V dissolution may also promote Ni passivation or secondary precipitation.

Kinetic Limitations and Mass Transfer Effects

Even when thermodynamically favorable, metal recovery from spent catalysts is often limited by kinetics. Surface passivation by residual sulfur, coke fragments, or reprecipitated oxides slows dissolution and results in incomplete extraction (Pathak et al., 2021; Zoraga, 2024). These effects are exacerbated when oxidation pretreatments form dense oxide shells around sulfide cores.

Diffusion constraints within catalyst pellets further limit reaction rates. Many spent catalysts retain their original extrudate geometry, with internal porosity collapsing during aging and regeneration (Hart, 2021). As a result, leaching kinetics are often governed by intraparticle diffusion rather than surface reaction rates, particularly for MoS_2 platelets embedded deep within the alumina matrix.

Attempts to overcome these barriers through aggressive grinding or intensified leaching often increase reagent consumption and secondary waste generation without proportional gains in recovery (Gao et al., 2024). From a critical perspective, kinetic limitations must be addressed through an integrated pretreatment–leaching design rather than isolated optimization of individual steps.

The thermodynamic stability fields of Ni, Mo, and V sulfides and oxides strongly influence the selection and sequencing of pretreatment and recovery routes. Differences in oxygen and sulfur potentials determine whether oxidation enhances metal accessibility or causes volatilization and losses. Table 4 summarizes the dominant stability domains of the three metals and highlights their direct implications for recycling strategies.

Table 4. Thermodynamic stability domains relevant to Ni–Mo–V systems. Adapted from Zhang et al. (2020), Le and Lee (2021).

Metal	Stable sulfide region	Oxide stability	Process implication
Ni	Low pO_2, high pS_2	NiO at high pO_2	Acid leach feasible
Mo	MoS_2 wide domain	MoO_3 volatile	Careful roasting
V	VS_x narrow	V_2O_5 stable	Oxidative routes

In conclusion, thermodynamic feasibility alone is insufficient to ensure effective recovery of Ni–Mo–V from spent catalysts. Stability boundaries, redox sensitivity, and diffusion-controlled kinetics together define narrow, process-specific operating windows. These constraints directly affect the viability of high-temperature routes, which are examined next in the section on Pyrometallurgical Recovery Routes.

Pyrometallurgical Recovery Routes

Pyrometallurgical processing is often presented as a robust shortcut for spent Ni–Mo–V catalysts, because it bypasses slow intraparticle leaching and directly concentrates metals into a reduced phase. This promise is only partially true. High-temperature routes can simplify solid handling and destroy organics, but they also introduce volatility losses, sulfur off-gas burdens, and weak selectivity. The objective of this section is to examine roasting, melting, and reduction-based enrichment critically, and to clarify where pyro routes add value and where they create new liabilities.

Incineration and Roasting (Sulfides → Oxides; Sulfur Management)

Oxidative roasting is commonly used to convert metal sulfides into oxides and to remove residual hydrocarbons. In principle, this step improves downstream leachability by transforming MoS_2, NiS_x, and VS_x into oxides that can be attacked in acid or alkaline media. In practice, roasting is inseparable from sulfur management. Sulfur is released mainly as SO_2, requiring capture and treatment systems that can dominate the environmental footprint and be part of the CAPEX.

Roasting also alters phase assemblages in ways that are not always beneficial. Vanadium and molybdenum can form complex oxides and mixed phases with alumina or silica, thereby reducing solubility and complicating separation. These risks increase when temperature control is poor or when the spent catalyst contains significant ash-forming impurities.

Figure 5 summarizes the main transformations expected during roasting and the points at which sulfur is released into the gas phase.

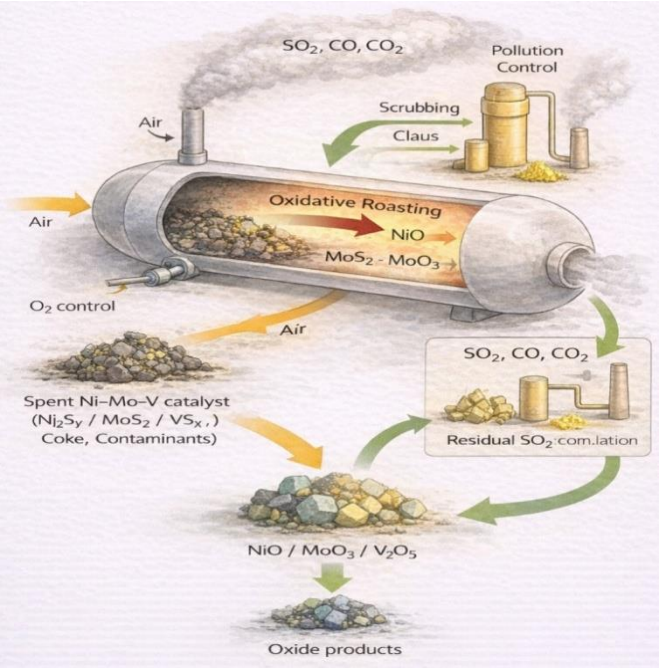

Figure 5. Conceptual pathway for oxidative roasting of spent Ni–Mo–V catalysts, highlighting sulfide-to-oxide conversion and SO_2 generation. Adapted from Shi et al. (2023).

The scheme emphasizes that roasting is not only a solid conditioning step but also an emissions-control problem that must be solved in parallel; otherwise, the overall route becomes environmentally fragile.

Smelting and Metallic Concentration (Benefits and Metal Losses)

Smelting routes concentrate Ni–Mo–V into an alloy or matte, called 'alloy enrichment.' High-temperature reduction produces an Ni–Mo–V–rich alloy for cleaner hydrometallurgical separation (Shi et al., 2023). This is useful when catalysts have high metal loadings, coke, or pore blockage, making leaching inefficient.

However, smelting is not selective. It focuses on what is reduced under the chosen conditions and can carry impurities into the alloy. More importantly, Mo and V are prone to losses into slag phases or volatile species depending on oxygen potential, slag chemistry, and temperature. These losses can offset the perceived advantage of "fast" concentration, particularly when slag capture and reprocessing are not engineered from the start.

From an environmental standpoint, smelting introduces two coupled issues: energy intensity and off-gas complexity. Any sulfur remaining after pretreatment can produce SO_2, while residual halogens or organics can complicate gas cleanup. These constraints can render pyrometallurgical options unattractive in jurisdictions with stringent air permitting requirements.

Table 5 contrasts typical outcomes of roasting- and smelting-based enrichment, focusing on metal distribution, sulfur control requirements, and downstream implications.

Table 5. Comparative advantages and risks of pyrometallurgical enrichment routes for spent Ni–Mo–V catalysts (roasting vs reduction–smelting). Adapted from Shi et al. (2023) and Ying et al. (2025).

Aspect	Oxidative roasting	Reduction–smelting
Main objective	Convert sulfides to oxides and remove coke	Concentrate metals into alloy or matte
Typical temperature range	400–700 °C	1,200–1,500 °C
Sulfur behavior	Released mainly as SO_2	Partitioned to slag and off-gas
Metal recovery potential	High for Ni and V; Mo sensitive	High for Ni; Mo and V losses possible
Selectivity	Moderate; depends on pO_2 control	Low; metals co-reduced
Energy demand	Moderate	High
Environmental risks	SO_2 emissions; need gas treatment	Slag disposal; high CO_2 footprint
Process control complexity	High (oxygen potential critical)	Moderate (thermal control dominant)
Downstream compatibility	Well suited for hydrometallurgy	Requires additional oxidation/leaching
Typical role in flowsheets	Pretreatment or upgrading step	Bulk metal concentration stage

Pyrometallurgy simplifies solid processing but increases complexity in gas handling and slag management, with uncertain selectivity for Mo and V.

Critical Appraisal (Energy, Selectivity, and Integration Needs)

A realistic appraisal must acknowledge that pyrometallurgical routes are rarely standalone solutions for Ni–Mo–V catalysts. Their strongest role is as an **enrichment step** that produces a smaller, more homogeneous intermediate. However, this advantage comes at the cost of high energy demand and limited chemical selectivity.

The most credible value proposition is therefore hybridization: use high-temperature treatment to remove organics and consolidate metals, then apply controlled hydrometallurgy for separation and purification. This logic is supported by routes in which an Ni–Mo–V alloy is first produced and then subjected to oxidation leaching and solvent extraction to selectively recover Mo and V selectively (Shi et al., 2024). Even in these hybrid chains, overall performance depends on controlling metal losses during enrichment and preventing impurity carryover, which would later poison solvent extraction.

Figure 6 outlines a representative hybrid route that uses pyrometallurgy to concentrate metals and hydrometallurgy to restore selectivity.

Figure 6. Hybrid pyro–hydro processing chain: alloy enrichment followed by oxidation leaching and solvent extraction for Mo/V separation. Adapted from Shi et al. (2024).

The hybrid logic addresses a core weakness of pyrometallurgy—low selectivity—while preserving its advantage in handling heavily fouled catalysts. The primary unresolved risks remain Mo/V losses during enrichment and the environmental burden of sulfur-bearing off-gases.

The next section examines Hydrometallurgical Recovery Routes, where selectivity is higher, but mass transfer, reagent demand, and residue management are the dominant constraints.

Hydrometallurgical Recovery Routes

Hydrometallurgy is the most selective and versatile route for recovering metals from spent Ni–Mo–V catalysts. Unlike pyrometallurgy, it allows decoupling of metal dissolution, separation, and purification under controlled chemical conditions. This section critically assesses the main hydrometallurgical strategies, their separation logic, and their practical limitations, with particular attention to sulfur behavior and process integration.

Overview of Hydrometallurgical Routes and Separation Logic

Most hydrometallurgical flowsheets exploit the contrasting chemical behavior of Ni, Mo, and V under acidic, alkaline, and oxidizing conditions. Mo and V form soluble oxyanions, whereas Ni dissolves as a divalent cation, enabling sequential or selective recovery (Le & Lee, 2021; Liang et al., 2022). This intrinsic selectivity explains why hydrometallurgy dominates industrial and pilot-scale recycling concepts (Wang et al., 2021b; Ying et al., 2025).

However, selectivity is conditional. Residual sulfur, coke-derived carbon, and secondary phases formed during pretreatment can distort expected behavior. Life-cycle and sustainability assessments also indicate that reagent consumption and effluent treatment often outweigh energy use as the primary drivers of environmental impact (Ibrahim et al., 2025; Kurniawan et al., 2024). These constraints must be considered when comparing routes.

Figure 7 illustrates the conceptual separation logic common across most hydrometallurgical flowsheets.

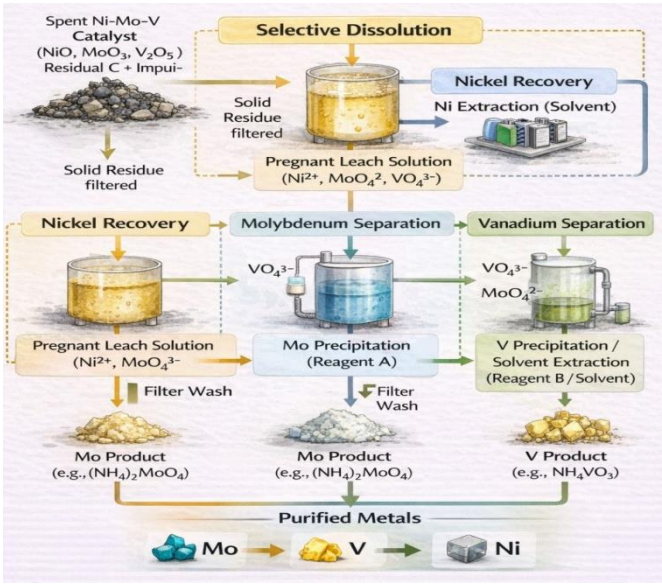

Figure 7. Generic hydrometallurgical separation logic for Ni–Mo–V spent catalysts, highlighting selective dissolution and downstream purification. Adapted from Le and Lee (2021) and Liang et al. (2022).

The scheme emphasizes that selectivity is achieved through sequential control of pH, redox potential, and complexation rather than through a single leaching step.

Mineral Acid Leaching and Stepwise Selective Processes

Mineral acid leaching remains the most established hydrometallurgical approach. Sulfuric and hydrochloric acids efficiently dissolve Ni, while Mo and V can be partially mobilized depending on oxidation state and acid strength (Cai et al., 2022; Chen et al., 2022). Stepwise leaching strategies exploit this behavior by first separating Ni, then recovering Mo and V from the residue or pregnant solution (Zhang et al., 2020).

Despite its maturity, mineral acid leaching has well-known drawbacks. High acid consumption, co-dissolution of alumina supports, and iron contamination complicate purification and increase waste volumes (Aslan et al., 2023; Xhaferaj & Ferella, 2022). From a critical standpoint, mineral acids are robust yet chemically aggressive tools that require tight control to maintain selectivity.

Organic Acids, Mechanochemistry, and Intensified Leaching

To mitigate the drawbacks of strong mineral acids, organic acids and intensified leaching techniques have attracted attention. Organic acids conditions and lower corrosion risk (Pathak et al., 2021). Mechanochemical activation further enhances metal accessibility by disrupting sulfide lattices and increasing defect density (Zoraga, 2024).

Ultrasound-assisted and ammoniacal leaching have also been proposed to overcome diffusion limitations and shorten reaction times (Saim, 2025; Şayan & Çalışkan, 2024). Although these approaches often show improved kinetics at the laboratory scale, their scalability and economic robustness remain uncertain. In many cases, efficiency gains are offset by greater operational complexity.

Alkaline and Oxidative Leaching Routes

Alkaline leaching is particularly effective for Mo and V recovery, as both metals form stable soluble species under basic and oxidizing conditions. Fenton-like and alkaline–oxidative systems can selectively mobilize V while leaving Ni largely undissolved (Lv et al., 2023). This selectivity makes alkaline routes attractive for multi-step flowsheets.

However, alkaline systems are sensitive to sulfur chemistry. Partial oxidation of sulfides can generate elemental sulfur (S^0), which may passivate surfaces or accumulate as fine solids. While S^0 formation can be advantageous for sulfur recovery, it also introduces handling and filtration challenges if not anticipated in the design.

Hydrometallurgical processing is key to recovering Ni–Mo–V hydroprocessing catalysts. The leaching medium affects metal selectivity, impurities, reagent use, and separation. As shown in Table 6, alkaline methods favor molybdenum and vanadium, while acidic methods are better for nickel but co-leach impurities. Organic and oxidative leaching add flexibility but come with kinetic and economic challenges.

Table 6. Comparison of major hydrometallurgical strategies. Adapted from Cai et al. (2022), Chen et al. (2022), Pathak et al. (2021).

Route	Target metal	Selectivity	Key challenge
Alkaline leach	Mo, V	High	Residues
Acid leach	Ni	Moderate	Impurities
Organic acids	Mo/V/Ni	Variable	Kinetics
Oxidative leach	V	High	Reagent cost

Solvent Extraction, Purification, and Product Routes

Solvent extraction (SX) is central to the production of product-grade Mo, V, and Ni streams. Organophosphorus extractants and tailored solvents enable selective separation of Mo and V from complex leach liquors (Bu et al., 2025; Pradhan et al., 2020). Subsequent precipitation or crystallization steps yield marketable products, including molybdic acid or vanadium salts (Leszczyńska-Sejda et al., 2023; Liu et al., 2024).

SX performance is highly sensitive to upstream leaching chemistry. Impurities, residual sulfur species, and organic degradation products can destabilize extraction systems (Xhaferaj & Ferella, 2022). Therefore, purification should be treated as an integrated component of the flowsheet, not a downstream afterthought.

Alternative, Electrochemical, and Modeling-Based Approaches

Electrochemical routes and redox-mediated extraction have been proposed as low-reagent alternatives that offer precise control over metal valence states (Xue & Wang, 2020). Although conceptually promising, these methods remain largely confined to proof-of-concept studies.

Process modeling and multiscale simulations are increasingly used to rationalize leaching behavior and optimize reagent use (Pudi, 2022). Although not recovery routes per se, these tools support more efficient flowsheet design and help bridge the gap between laboratory results and industrial applications.

Oxidative Leaching and Sulfur as a Recoverable Phase

Oxidative leaching can convert sulfide sulfur to elemental sulfur rather than to SO_2. This pathway enables the recovery of sulfur as a solid by-product rather than treating it solely as an emission. When integrated thoughtfully, such systems can align metal recovery with sulfur management strategies, including downstream Claus-based conversion routes.

From a critical perspective, the success of hydrometallurgy lies not in any single leaching chemistry but in the coherent

integration of dissolution, purification, and sulfur control. These integration challenges are addressed explicitly in the next section, "Integrated Metal and Sulfur Recovery."

Integrated Metal and Sulfur Recovery

Sulfur management is often treated as an auxiliary environmental obligation in spent catalyst recycling. This section adopts a different perspective: sulfur is treated as a recoverable resource whose behavior must be integrated into metal-recovery strategies. The objective is to critically assess sulfur-handling routes and identify synergies between metal extraction and sulfur valorization.

H_2S Purification, Upgrading, and Sulfur as a Resource

Sulfur-bearing streams generated during pretreatment, roasting, or leaching pose regulatory challenges and recovery opportunities. Technologies for H_2S purification and chemical upgrading have evolved beyond simple abatement, aiming to recover sulfur or transform it into value-added intermediates (Jiang & Li, 2025). In spent catalysts, sulfur originates primarily from metal sulfides and residual hydrocarbons, making the controlled conversion of sulfur central to process sustainability.

Treating sulfur as a recoverable material rather than an emission shifts flowsheet priorities. Instead of minimizing sulfur formation, integrated routes aim to concentrate sulfur into manageable streams, preferably as elemental sulfur. This approach reduces the environmental burden of off-gas treatment and improves overall resource efficiency.

Claus-Related Catalyst Handling and Performance Limitations

The Claus process remains the industrial benchmark for converting H_2S to elemental sulfur. However, integrating it with catalyst recycling is challenging. Sulfur-bearing off-gases from spent catalyst treatment may contain contaminants that degrade Claus catalyst performance or accelerate deactivation (Fallah et al., 2020). Moreover, spent Claus catalysts themselves become secondary wastes that require treatment or regeneration.

Modifications to Claus catalysts, including metal oxide doping, have been proposed to improve sulfur yield and resistance to poisoning (Sui et al., 2020). These improvements are relevant to the design of integrated schemes, enabling sulfur recovery units to tolerate variable feed compositions typical of recycling operations.

Figure 8 shows how sulfur-bearing streams from catalyst recycling can be routed to Claus-type recovery units.

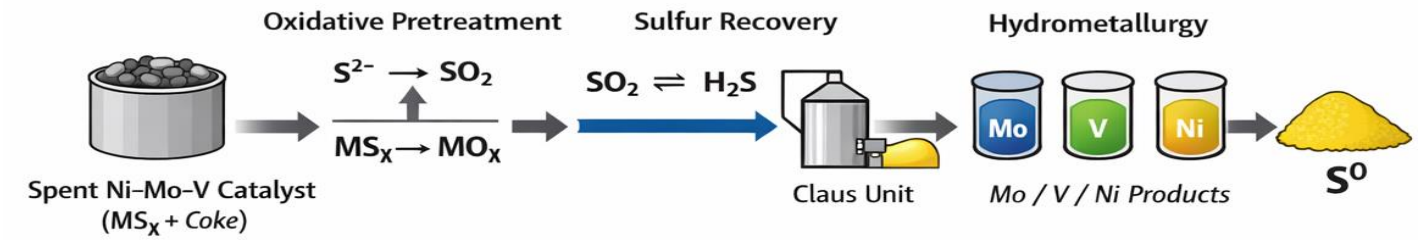

Figure 8. Conceptual integration of spent catalyst pretreatment with Claus-based sulfur recovery. Adapted from Fallah et al. (2020) and Sui et al. (2020).

The scheme highlights that sulfur recovery efficiency depends not only on H_2S concentration but also on the chemical cleanliness of the gas stream supplied to the Claus reactor.

Low-Temperature Sulfur Recovery and Selective Oxidation Concepts

To address the high-temperature Claus system limits, low-temperature sulfur recovery methods such as liquid-phase sulfur recovery and selective oxidation have been proposed. They enable sulfur formation under milder conditions, reducing energy use and catalyst degradation (Prinsloo et al., 2021; Sun et al., 2022).

Heterogeneous catalytic oxidation of H_2S provides flexibility for dilute or fluctuating sulfur streams (Lee, 2021; Kraia et al., 2023). While not yet widely used industrially, these methods are useful for hydrometallurgical circuits in which sulfur is released gradually rather than as a concentrated gas.

From a critical standpoint, low-temperature routes reduce thermal stress and emissions but often face challenges related to catalyst stability and sulfur-separation efficiency. Therefore, their role is complementary rather than substitutive to Claus-based systems.

Sulfur-Containing Streams as Operational Analogs

Insights into sulfur management can be gained from biogas upgrading and oxidative methane coupling. Studies of sulfur-containing biogas show how trace H_2S and NH_3 impact catalyst stability and reaction pathways (Gu et al., 2021; Gao et al., 2023). These systems help understand sulfur poisoning, regeneration, and long-term operation.

Applying these lessons to spent-catalyst recycling underscores the need for early sulfur control. Whether sulfur is removed before metal recovery (sulfur-first) or recovered downstream (metal-first), its behavior influences catalyst lifetime, separation efficiency, and waste generation.

Integrated Flowsheets and Process Synergies

Integrated metal–sulfur flowsheets can be divided into two main approaches. Metal-first treats sulfur as off-gas or residue after metal extraction, while sulfur-first converts and recovers sulfur before metal leaching. Each involves trade-offs in complexity, selectivity, and environmental impact.

Hybrid pyro–hydro flowsheets offer the greatest potential for synergy. High-temperature steps destroy organics and concentrate sulfur in streams, while hydrometallurgy provides selectivity for metal separation. When properly integrated, these systems reduce final waste volumes and enable near-complete valorization of spent catalyst.

Efficient sulfur management is vital in spent catalyst recycling because sulfur species impact metal recovery and environmental performance. During processing, sulfur may appear as sulfides, SO_2, H_2S, or elemental sulfur, needing specific conversion and capture methods. Table 7 lists the main sulfur recovery approaches reported in the literature, highlighting their roles in metal–sulfur recovery schemes.

Table 7. Metal-first vs sulfur-first integrated strategies. Adapted from Jiang and Li (2025), Prinsloo et al. (2021).

Strategy	Strength	Limitation
Metal-first	Maximizes recovery	Sulfur emissions
Sulfur-first	Emission control	Metal dilution
Integrated	Balanced performance	Process complexity

Low-temperature and integrated sulfur-recovery routes outperform traditional oxidation for emissions control and resource efficiency. Valorizations that produce elemental S^0 reduce waste and support circular economy goals. Effective implementation requires tight process integration, gas control, and compatibility with catalyst pretreatment, highlighting the need for coordinated sulfur and metal recovery planning.

In summary, sulfur management is not peripheral to catalyst recycling. It is a central design variable that determines environmental compliance, process efficiency, and circularity. These integration challenges motivate the discussion in the next section on Emerging and Alternative Technologies, which examines novel concepts to further improve metal–sulfur co-recovery.

Emerging and Alternative Technologies

This section discusses emerging technologies that may complement or reshape conventional metal and sulfur recovery routes. The objective is not to overstate readiness but to critically assess the realistic contributions, limitations, and relevance of these approaches to the recycling of spent Ni–Mo–V catalysts.

Emerging solvent systems and bio-assisted routes aim to improve selectivity and sustainability in hydrometallurgical recycling of spent catalysts by reducing reagent use, enhancing metal discrimination, and lowering environmental impact, despite facing scale-up and economic challenges. Table 8 summarizes their key features, advantages, and limitations.

Table 8. Emerging technologies: maturity and applicability. Adapted from Srivastava et al. (2025), Paiva & Nogueira (2021).

Technology	Target	TRL	Main bottleneck
Bioleaching	Mo, V	Low–medium	Toxicity
Ionic liquids	Critical metals	Low	Cost
Plasma	Pretreatment	Low	Scale
DES	Selective leach	Low	Stability

The comparison shows bio-assisted and non-conventional solvents can improve selectivity and reduce chemical use under controlled conditions, but their practical use is limited by slow kinetics, stability issues, and high costs. These methods are better as niche or complementary options than standalone industrial routes, highlighting the value of hybrid processes combining mature hydrometallurgy with targeted intensification.

Figure 9 summarizes alternative approaches for recycling spent Ni–Mo–V catalysts, highlighting their role, TRL, and potential for integration in established flowsheets. These technologies are not yet mature recovery pathways.

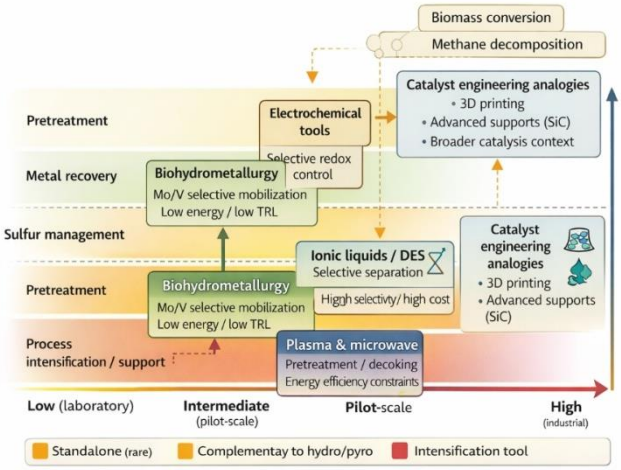

Figure 9. Emerging and alternative technologies for recycling spent Ni–Mo–V catalysts. Adapted from Srivastava et al. (2025), Hocheng et al. (2025), Moosakazemi et al. (2023), Paiva and Nogueira (2021), Adebayo (2025), Králik et al. (2025), Tuci et al. (2021), Ahmad et al. (2025), Ubaid et al. (2025), Pehlivan et al. (2021), Anderson (2022), Lycourghiotis and Kordouli (2025), and Cao et al. (2020).

Biohydrometallurgy and Bioleaching for Secondary Resources

Biohydrometallurgy has gained attention as a low-energy alternative for metal recovery from secondary resources. For spent Ni–Mo–V catalysts, biological systems show the greatest potential for mobilizing Mo and V, particularly

through indirect bioleaching mechanisms involving ferric iron or biogenic oxidants (Srivastava et al., 2025; Hocheng et al., 2025).

However, nickel recovery remains limited. Ni dissolution is often slow and incomplete because of strong associations with alumina supports and passivating sulfur layers. Toxicity of spent catalysts, variable sulfur chemistry, and long residence times further constrain scalability (Moosakazemi et al., 2023). As a result, biohydrometallurgy is better viewed as a niche or preconditioning option rather than a standalone solution for Ni–Mo–V systems.

Ionic Liquids and Advanced Solvents

Ionic liquids and deep eutectic solvents have been proposed as selective media for extracting critical metals from urban and industrial wastes. Their tunable polarity and complexation behavior offer theoretical advantages for the separation of Mo and V under mild conditions (Paiva & Nogueira, 2021).

Despite promising laboratory results, practical barriers persist. High solvent costs, viscosity-related mass-transfer limitations, and sensitivity to impurities hinder large-scale adoption. For spent catalysts, ionic liquids are most relevant as specialty separation tools or for treating concentrated intermediates, rather than for treating bulk solids.

Assisted Processes: Microwave and Cold Plasma

Process intensification using microwave heating or cold plasma aims to overcome kinetic barriers without excessive thermal input. Microwave-assisted leaching can enhance reaction rates by selectively heating sulfide phases, while cold plasma can disrupt coke and sulfur species at low bulk temperatures.

Yet these approaches face scale-up and energy-efficiency challenges similar to those observed in plasma regeneration of catalysts. Their greatest potential lies in targeted applications, such as selective pretreatment or regeneration, rather than in full recycling flowsheets.

3D Printing, Advanced Supports, and Catalyst Engineering Analogies

Advances in catalyst engineering offer useful analogies for recycling. Developments in 3D-printed catalysts and structured supports highlight how geometry and mass-transfer control influence reaction efficiency (Adebayo, 2025; Králik et al., 2025). These concepts can inform the design of recycling reactors and leaching systems.

Similarly, studies of silicon carbide (SiC) supports highlight chemical and thermal stability under harsh conditions (Tuci et al., 2021). Although not directly applied to recycling, these materials demonstrate how support selection governs durability and process integration in sulfur-rich environments.

Catalytic Process Analogies: Methane Decomposition and Oxidative Desulfurization

Research on methane decomposition, oxidative desulfurization, and advanced catalyst design provides indirect insights into sulfur tolerance and regeneration strategies (Ahmad et al., 2025; Ubaid et al., 2025; Pehlivan et al., 2021). These studies underscore the importance of controlling sulfur–metal interactions and preserving active surface accessibility.

Although these systems are not recovery processes, they inform understanding of sulfur poisoning, regeneration limits, and long-term catalyst behavior relevant to recycling.

Broader Catalysis Context: Biomass Conversion and Sustainable Fuels

Trends in biomass conversion and sustainable fuel production indicate that catalysis research is increasingly integrating sustainability, material efficiency, and lifecycle thinking (Anderson, 2022; Lycourghiotis & Kordouli, 2025; Cao et al., 2020). These developments reinforce the shift toward circular approaches, in which catalysts are designed with end-of-life recovery in mind.

In summary, emerging technologies offer valuable concepts and targeted tools, but few are ready to replace established pyro–hydro routes for spent Ni–Mo–V catalysts. Their primary contributions lie in hybridization, selective pretreatment, and a deeper understanding of sulfur–metal interactions. These perspectives set the stage for the next section on Industrial Practices and Patents, which examines technological maturity and real-world constraints.

Industrial Practices, Techno-Economic Feasibility, and Cross-Sector Integration

This section bridges laboratory-scale routes and industrial reality. The objective is to assess feasibility, scale-up constraints, and how recycling spent Ni–Mo–V catalyst aligns with existing refinery operations, patents, and adjacent waste streams.

Techno-Economic Feasibility and Plant-Level Constraints

Industrial implementation depends on CAPEX/OPEX trade-offs, reagent use, residue management, and product quality. Techno-economic analyses indicate that pretreatment and purification costs predominate, whereas logistics and permitting constraints constrain throughput (Marafi et al., 2024; Pathak et al., 2021). Scale-related selectivity losses, such as Mo/V volatility or Ni passivation, increase solvent demand and effluent loads. Flowsheets that minimize the number of unit operations and reuse reagents outperform complex, highly selective options.

Table 9 compares emerging and alternative methods for recycling spent Ni–Mo–V catalysts, highlighting their roles, maturity, and limitations in complex refinery applications. It emphasizes distinguishing practical, industry-ready solutions from lab-based ideas.

Table 9. Comparative techno-economic drivers for hydrometallurgical vs hybrid pyro–hydro routes for spent Ni–Mo–V catalysts (CAPEX drivers, OPEX sensitivities, residue generation). Adapted from Marafi et al. (2024) and Pathak et al. (2021).

Aspect	Hydrometallurgical routes	Hybrid pyro–hydro routes
Main CAPEX drivers	Reactors, solid–liquid separation units, solvent extraction circuits	High-temperature furnaces, off-gas treatment, plus downstream leaching units
CAPEX sensitivity	Increases with the number of separation and purification stages	Dominated by furnace size, refractory lining, and gas handling
Main OPEX drivers	Reagents (acids/alkalis), extractants, water consumption	Energy demand, reductants, fluxes, gas treatment
OPEX sensitivity	Highly sensitive to reagent price and recycling efficiency	Highly sensitive to energy cost and operating temperature
Process selectivity	High (metal-specific leaching and SX)	Low during smelting; selectivity restored in hydro stage
Residue generation	Leach residues, gypsum/neutralization solids	Slag generation plus secondary leach residues
Residue management cost	Moderate; depends on residue stability	Higher due to slag handling and disposal
Operational flexibility	High; modular and adaptable	Lower; constrained by furnace operation
Typical TRL	Medium to high (pilot–industrial)	Medium (industrial pyro + pilot hydro)
Overall economic robustness	Strong for selective, metal-focused recovery	Justified mainly for complex feeds or high metal grades

Most emerging technologies are at low to intermediate readiness levels and unlikely to replace traditional recovery methods soon. Biohydrometallurgy offers benefits in selectivity and environmental impact but is slow and limited to nickel-rich phases. Ionic liquids and deep eutectic solvents are efficient under controlled conditions, but their high cost, recyclability issues, and scale-up problems hinder industrial use. Process-assisted techniques such as plasma or electrochemical methods are better viewed as tools for process enhancement rather than as primary recovery methods. Overall, these technologies serve as supporting elements in integrated recycling strategies, emphasizing the need for hybrid process designs over single-route solutions.

Industrial-Scale Product Route Example: Molybdic Acid

Industrial recovery of molybdic acid from spent catalysts exemplifies a viable end-product pathway. Scale-up relies on controlled oxidation, selective leaching, and crystallization to meet purity standards while managing vanadium co-extraction (Leszczyńska-Sejda et al., 2023). This route shows that product-driven design—rather than metal-by-metal maximization—improves economic robustness.

Related Refinery/Industrial Waste Streams and Integration Opportunities

Operational synergies exist among refinery wastes that share sulfur, alkalinity, or metal-bearing characteristics. Spent caustic and refinery wastewater treatments share parallels in oxidation control, neutralization, and sulfur management (Mosleh et al., 2025; Zhang et al., 2023; Munoz et al., 2020). Beyond refineries, tailings, mattes, and iron tailings present logistics and separation challenges analogous to those in spent catalyst recycling (Gray et al., 2023; Xiao et al., 2021; Wu et al., 2024).

Figure 10 shows system integration opportunities in refinery operations, highlighting shared environments, sulfur handling, and pH adjustment across spent catalyst recycling, caustic treatment, and wastewater systems. This approach supports decarbonization and waste reduction by reducing chemical use and sulfur residues.

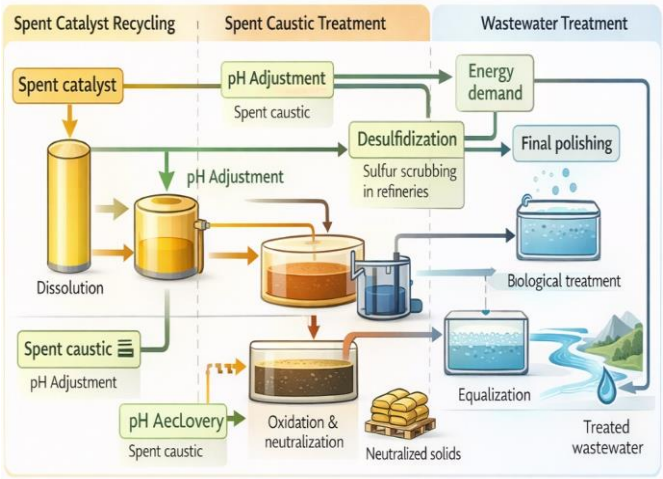

Figure 10. Integrated refinery waste management highlighting coupling points between spent catalyst recycling, spent caustic treatment, and wastewater systems. Adapted from Mosleh et al. (2025), Zhang et al. (2023), and Munoz et al. (2020).

The integrated layout identifies key convergence points: sulfur streams from catalyst pretreatment or regeneration can be routed to existing desulfurization units, reducing infrastructure duplication. Alkaline spent caustic streams may neutralize acidic effluents if the impurities are compatible. Biological polishing and equalization basins serve as final barriers to residual sulfur and trace metals.

Such integration isn't inherently beneficial because of variability in catalysts, sulfur loadings, and toxic metals (Ni, V, Mo), which require strict control. Without proper monitoring and segregation, cross-contamination risks may negate circular benefits. Thus, integration needs mass-balance modeling, speciation analysis, and regulatory checks before industrial use.

Benchmarks from Precious-Metal Recovery and Hazardous Wastes

Precious-metal recovery from converters sets benchmarks for selectivity, value density, and separation rigor, underscoring the need for Ni–Mo–V systems to prioritize product quality and market alignment (Adelekun, 2023; Moleko-Boyce et al., 2022; McCarthy et al., 2021). A broader hazardous-waste management framework highlights compliance, stabilization, and long-term liability as decisive factors (Mandal et al., 2023; Marcelino et al., 2025).

Circular Strategies and Methodological Comparators

Cross-sector circular strategies link catalyst recycling with batteries, balancing risk and infrastructure sharing (Garole et al., 2020). Reviews on cobalt secondary resources offer methodological comparators for scale-up and LCA (Chandra et al., 2022). Deactivation analogies from tar-reforming catalysts highlight the need for sulfur and coke control (Mohamed, 2022). Urban mining frameworks support the integration of spent catalysts into resource recovery systems (Ilyas et al., 2021).

Spent Ni–Mo–V catalysts can be integrated into a broader circular economy beyond refineries, connecting with waste streams like refinery residues, tailings, batteries, and metal residues. Figure 11 shows these linkages and material flows that go beyond single-industry recycling. The goal is to find shared recovery methods and integration opportunities without implying direct interchangeability.

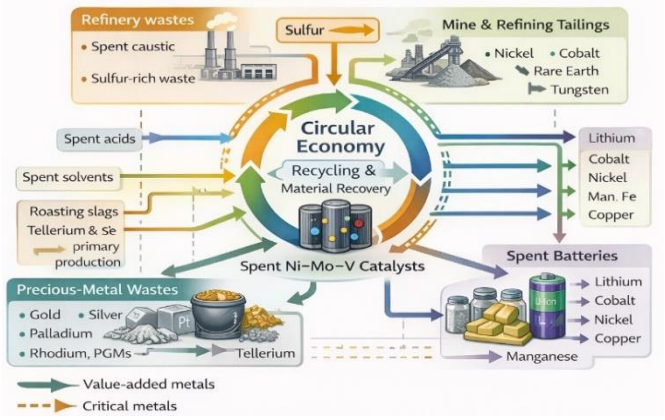

Figure 11. Cross-sector integration map linking spent Ni–Mo–V catalysts with refinery wastes, tailings, batteries, and precious-metal streams within a circular-economy framework. Adapted from Garole et al. (2020) and Ilyas et al. (2021).

The integration map shows spent catalysts as a middle ground between waste management and critical-metal recovery. Nickel and molybdenum flows overlap with battery recycling, while vanadium recovery intersects with steel and tailings strategies. Precious metals serve as benchmarks for separation and recovery, despite varied compositions.

Cross-sector integration faces constraints from differences in impurity profiles, regulations, and scale. Tailings and battery streams require distinct pretreatment and separation methods. Integration should focus on infrastructure, solvent systems, and recovery platforms rather than direct co-processing without adaptation. From a circular-economy perspective, spent Ni–Mo–V catalysts are hazardous waste and a secondary resource in metal-recovery ecosystems.

These practices and pathways frame the non-technical constraints that shape deployment. The next section covers Environmental, Health, and Regulatory Aspects, which influence permitting, ESG, and long-term operations.

Environmental, Health, and Regulatory Aspects

This section critically examines the environmental and health implications of spent Ni–Mo–V catalysts, positioning regulatory compliance and sustainability metrics as decisive factors for technology selection and scale-up.

Hazardous Waste Classification and Management Context

Spent hydroprocessing catalysts are widely classified as **hazardous waste** due to their content of heavy metals, sulfides, residual hydrocarbons, and potential for self-heating and toxic gas release. Regulatory frameworks in Europe, North America, and Asia consistently classify these materials as hazardous waste, imposing strict requirements for handling, transport, and treatment (Rosli et al., 2023; Mandal et al., 2023; Papanikola et al., 2021). Improper storage or pretreatment can lead to spontaneous oxidation, SO_2 and H_2S emissions, and metal leaching into soil and groundwater (Fu et al., 2021).

From a management perspective, stabilization and controlled regeneration are often mandated prior to any recovery step. These requirements increase operational costs but also create incentives for on-site or refinery-integrated recycling solutions that minimize off-site transport and long-term liability.

Ecotoxicity and Comparative Hazard Evidence

Ecotoxicological studies on refinery catalysts, particularly FCC analogs, provide comparative insight into environmental risks associated with spent Ni–Mo–V materials. Chronic toxicity to aquatic organisms has been linked to metal leaching and dispersion of fine particulates,, even after partial stabilization (Wang et al., 2021a). Similar hazard profiles are reported for hydroprocessing catalysts when sulfur and metal speciation are not adequately controlled during treatment (Fu et al., 2021).

These findings reinforce that recovery routes cannot be evaluated solely on metal yield. Emissions control, residue inertness, and long-term ecotoxicity must be considered integral performance metrics, especially for processes involving high-temperature oxidation or aggressive leaching.

Table 10 summarizes key hazard-related aspects of spent Ni–Mo–V hydroprocessing catalysts, framing environmental, health, and regulatory factors that influence recycling route selection and design. These aspects are presented as intrinsic process drivers affecting pretreatment, recovery technology choice, and residue management, not just regulatory compliance.

Table 10. Environmental, health and regulatory aspects associated with spent Ni–Mo–V catalysts and recovery routes. Adapted from Fu et al. (2021), Wang et al. (2021a), Rosli et al. (2023), and Papanikola et al. (2021).

Aspect	Spent Ni–Mo–V catalysts	Implications for recovery routes	Regulatory relevance
Hazardous classification	Heavy metals, sulfides, hydrocarbons	Mandatory pretreatment and stabilization	Hazardous waste regulations
Gas emissions	SO_2, H_2S during oxidation	Need for off-gas treatment and sulfur control	Air quality standards
Metal toxicity	Ni, V, Mo leachability	Residue inertization required	Soil and water protection
Ecotoxicity	Chronic aquatic toxicity	Limits on residue disposal	Environmental licensing
Residue generation	Solid residues, sludges	Trade-off between recovery yield and waste	Landfill restrictions
Carbon footprint	Energy-intensive steps	Route-dependent GHG intensity	ESG and LCA reporting

The comparison shows that hazardous classification, gas emissions, and metal toxicity are interconnected factors affecting recovery routes. Sulfide oxidation requires SO_2 and H_2S control, while Ni, Mo, and V leachability influence residue stabilization and disposal. Ecotoxicity and residue production create trade-offs between recovery and waste reduction. These aspects emphasize the importance of integrated environmental control, early regulatory alignment, and lifecycle-focused decisions in recycling spent Ni–Mo–V catalysts.

To synthesize the environmental and regulatory dimensions discussed above, Figure 12 maps the primary risk pathways and sustainability trade-offs associated with various spent Ni–Mo–V catalyst recycling routes, highlighting how process choices affect emissions, residues, and life cycle performance.

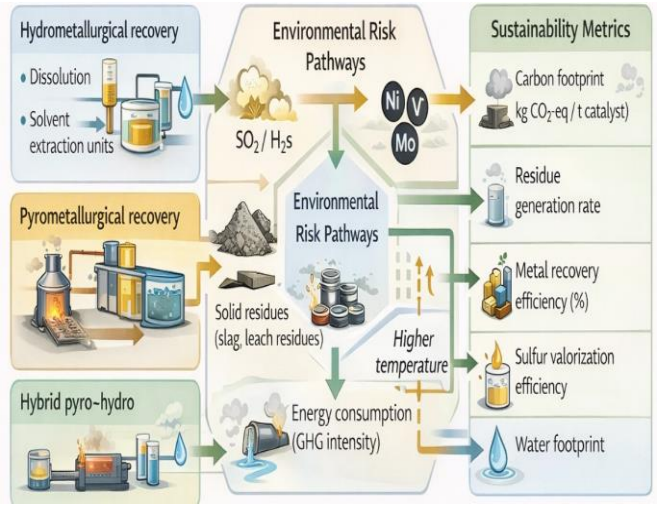

Figure 12. Environmental risk pathways and sustainability trade-offs associated with hydrometallurgical, pyrometallurgical, and hybrid recycling routes for spent Ni–Mo–V catalysts. Adapted from: Marafi et al. (2024); Aromaa-Stubb et al. (2025); Baritto et al. (2025); Munoz et al. (2020).

Figure 12 outlines environmental risks associated with hydrometallurgical, pyrometallurgical, and hybrid recycling of spent Ni–Mo–V catalysts, and links them to sustainability. Key environmental burdens include gas emissions (SO_2, H_2S), solid residues, and energy demand, affected by temperature and separation methods. Benefits of integrated processes are seen in recovery efficiency, sulfur valorization, and water footprint. Optimized hybrid routes can reduce environmental impact by balancing energy use with improved metal recovery and sulfur management, promoting a circular economy over single-metric focus.

Circular Economy Framing and Sustainability Metrics

Circular Economy and Urban Mining Perspective

Spent Ni–Mo–V catalysts are urban ores with metal concentrations often exceeding primary deposits. Circular economy frameworks view catalyst recycling as a means to reduce dependence on virgin mining, enhance resource security, and lower environmental impacts (Ippolito et al., 2023; Moita Neto et al., 2025; Ilyas et al., 2021). In places like Brazil, urban mining focuses on integrating with existing industry to overcome logistical and economic hurdles (Moita Neto et al., 2025; Funari et al., 2023).

Reciprocal urban mining concepts further advocate for system-level optimization, in which waste streams are dynamically routed to maximize overall value rather than isolated recovery efficiency (Song et al., 2025). Ionic liquids and bio-based approaches are often discussed in this context, although their large-scale sustainability remains case-dependent (Paiva & Nogueira, 2021).

Life Cycle Assessment and GHG Comparisons

Life cycle assessments show metal recovery from spent catalysts leads to much lower greenhouse gas emissions than primary mining, especially for Mo and V (Amato et al., 2025; Aromaa-Stubb et al., 2025). GHG savings are greatest with hybrid or hydrometallurgical methods that avoid high-temperature smelting and enable reagent recycling (Baritto et al., 2025). However, outcomes depend on system boundaries. Including pretreatment energy, effluent treatment, and sulfur management can offset benefits if not optimized (Munoz et al., 2020).

Critically, LCA results indicate that no single route is universally superior. Environmental performance depends on integration level, energy mix, and product slate. These findings underscore the need to align process selection with site-specific constraints and regulatory expectations rather than rely on generic sustainability claims.

Environmental and regulatory factors set feasibility thresholds, but technology choice relies on economic viability and maturity. The next section presents a Techno-Economic and TRL Assessment that summarizes the performance, cost, and readiness of recovery routes.

Techno-Economic and TRL Assessment

This section consolidates the technical maturity, economic drivers, and scale-up readiness of the primary recovery routes for spent Ni–Mo–V catalysts. The objective is not to rank routes by laboratory performance but to compare recoverable value, complexity, and deployment feasibility under industrial constraints.

Comparative Techno-Economic and TRL Perspective

Techno-economic assessments consistently show that hydrometallurgical routes excel in selectivity and flexibility, whereas pyrometallurgical routes offer robustness at the expense of energy intensity and metal losses (Marafi et al., 2024; Pathak et al., 2021; Ying et al., 2025). Hybrid pyro–hydro chains aim to balance these trade-offs by concentrating metals thermally and selectively separating them downstream (Shi et al., 2024).

Technology readiness levels (TRL) vary widely. Conventional roasting–leaching flowsheets and mineral-acid leaching are already deployed industrially, whereas biohydrometallurgy, ionic liquids, and electrochemical routes remain at the laboratory or pilot scale (Le & Lee, 2021; Srivastava et al., 2025). Importantly, higher recovery yields do not necessarily translate to better economics, as CAPEX, reagent recycling, and effluent treatment often dominate total costs.

Table 11 compares recovery routes for spent Ni–Mo–V catalysts, including metal recovery, TRL, and cost profiles. Values are typically ranges from literature and industry, noting variability from feed, pretreatment, and integration.

Table 11. Comparison of recovery efficiency, technology readiness level (TRL), and relative economic indicators for major spent Ni–Mo–V catalyst recycling routes. Adapted from Le and Lee (2021), Zhang et al. (2020), Shi et al. (2024), Pathak et al. (2021), Marafi et al. (2024), Srivastava et al. (2025), and Ying et al. (2025).

Route	Main metals recovered	Typical recovery (%)	TRL	CAPEX (relative)	OPEX (relative)
Roasting + acid leaching	Ni, Mo, V	70–90	8–9	Medium–High	High
Hybrid pyro–hydro (alloy + leach/SX)	Ni, Mo, V	75–95	6–7	High	Medium
Mineral acid leaching (stepwise)	Ni, Mo, V	80–95	7–8	Medium	Medium–High
Organic acid / intensified leaching	Mo, V (selective)	60–85	4–6	Low–Medium	Medium
Alkaline / oxidative leaching	Mo, V	70–90	6–7	Medium	Medium
Biohydrometallurgy	Mo, V	40–70	3–5	Low	Low
Ionic liquids / DES	Mo, V, Ni (selective)	60–90	3–4	High	High

The comparison shows that higher recovery efficiencies are linked to increased process complexity and cost. Roasting and mineral acid methods have high TRL but require significant energy and off-gas management. Hybrid pyro–hydro methods improve selectivity and alloy enrichment but require greater capital investment. Emerging methods such as organic acids, biohydrometallurgy, and ionic liquids offer lower environmental impact or higher selectivity, yet face scale-up and cost challenges. These differences highlight the importance of assessing recovery routes in a techno-economic and environmental context, not just in terms of extraction efficiency.

Critical Interpretation

From a critical standpoint, industrially viable routes are in TRL 7–9, where simplicity and compliance outweigh metal

recovery gains. Emerging routes are promising for selectivity and sustainability but lack proven economic competitiveness at scale. TRL advancement depends more on integration, residue reduction, and refinery compatibility than on chemistry innovations.

Techno-economic and maturity constraints reveal unresolved challenges hindering wider deployment. The next section discusses Knowledge Gaps and Research Needs, highlighting where future efforts should focus to bridge the gap between laboratory innovation and industrial implementation.

Knowledge Gaps and Research Needs

This section synthesizes the key limitations that still prevent broadly replicable, low-risk implementation of spent Ni–Mo–V catalyst recycling. The goal is to define a research agenda that is technically grounded and aligned with industrial constraints, rather than proposing incremental laboratory variations with limited scale relevance.

Full Metal–Sulfur Integration Remains Underdeveloped

Most flowsheets treat sulfur as a secondary constraint, managed through off-gas handling or neutralization, not as a co-product or design variable. This gap is critical because sulfur speciation affects leaching, residue stability, and emissions. Reviews focus on metal separation but rarely quantify how sulfur decisions alter process feasibility (Le & Lee, 2021; Liang et al., 2022; Wang et al., 2021b; Ying et al., 2025). An urgent need is an integrated metal–sulfur mass-balancing approach linking pretreatment to recovery and valorization options.

Low-Emission Pathways Need Comparable Performance Metrics

Low-emission claims are often qualitative and lack comparable metrics across routes. For instance, hybrid and hydrometallurgical routes may reduce energy demand relative to smelting but can increase effluent treatment loads and generate secondary residues. Techno-economic studies show that "cleaner" routes can become non-competitive once wastewater treatment and compliance costs are internalized (Marafi et al., 2024). Future work should adopt standardized indicators that jointly report: (i) recovery yields and purity, (ii) gaseous emissions (SO_2/H_2S), (iii) effluent load, and (iv) residue inertness, using consistent system boundaries (Le & Lee, 2021; Ying et al., 2025).

Modularization and Scalable Unit Operations Are Rarely Addressed Explicitly

Most lab studies optimize small-scale chemistry but do not translate unit operations into scalable, modular process blocks. This gap exists because industrial deployment is often incremental, limited by footprint, permitting, and utility integration. Reviews identify routes but fewer include scale-up logic for mixing, solid–liquid handling, filtration, SX circuit design, or reagent recycle (Liang et al., 2022; Wang et al., 2021b). Research should focus on pilot-relevant modular designs like skid-mounted pretreatment, leach–SX trains, and closed-loop sulfur/acid handling.

Complex and Heterogeneous Catalysts Remain the Hardest Industrial Case

Industrial catalyst waste is rarely uniform due to feed variability, multi-metal contamination, mixed supports, and variable coke/oil loading, causing inconsistent performance. Biohydrometallurgical approaches and low-temperature sulfur recovery are promising but their robustness to heterogeneity remains unestablished (Srivastava et al., 2025; Jiang & Li, 2025). Future research should go beyond "single-catalyst" tests to include: (i) mixed catalyst lots, (ii) high-contaminant cases, and (iii) realistic oil and sulfur loads, evaluating product quality and residue stability.

These gaps show progress relies on integrated design choices, not isolated leaching or extraction improvements. The Next section, Future Perspectives, describes development pathways and scenarios aligning new technologies with industry constraints.

Future Perspectives

This section outlines plausible development pathways for recycling spent Ni–Mo–V catalysts, grounded in industrial reality and regulatory momentum rather than speculative chemistry. The perspective adopted here emphasizes system integration, compliance-driven innovation, and digital enablement as the dominant forces shaping future deployment.

Spent Catalysts as Strategic Secondary Resources

Spent hydroprocessing catalysts are gaining importance as secondary resources because they contain high, predictable levels of Ni, Mo, and V compared to primary ores. Supply chain issues, geopolitical risks, and lengthy mine development increase the relevance of catalyst-derived metals as short-term supplements to primary production (Le & Lee, 2021; Liang et al., 2022; Ying et al., 2025). Thus, catalyst recycling moves from waste management to a key resource-security strategy within refineries.

Circular Economy Implementation in Refining

Circular economy principles in refining shift from concept to closing material loops. For spent Ni–Mo–V catalysts, this means metal recovery, sulfur valorization, reagent recycling, and inertization within refineries (Ippolito et al., 2023; Moita Neto et al., 2025). Integrated flowsheets that co-process spent catalysts with caustic, wastewater, or sulfur streams are gaining traction to reduce logistics, complexity, and environmental liabilities (Munoz et al., 2020; Ilyas et al., 2021).

Regulatory Pressure as a Driver of Technological Selection

Environmental regulation now drives innovation, not constraints. Stricter limits on SO_2/H_2S emissions, waste disposal, and lifecycle carbon favor recovery methods that reduce off-site treatment and residues (Fu et al., 2021; Mandal et al., 2023). As LCA and ESG reporting become mandatory, technologies with transparent, auditable performance in emissions, residues, and recovery will outperform those focused only on metal yield (Amato et al., 2025; Aromaa-Stubb et al., 2025; Baritto et al., 2025).

Digitalization and Advanced Process Control

Digitalization is an underutilized lever in catalyst recycling. Advanced sensors, real-time gas monitoring (SO_2/H_2S), and control of oxidation and leaching can reduce variability caused by feed heterogeneity. With digital twins and data-driven optimization, these tools enable adaptive operation, improved environmental control, and faster scale-up (Ying et al., 2025; Le & Lee, 2021). Future plants will rely on digital control as much as chemical selectivity for performance.

Looking beyond current tech, Table 12 shows external drivers shaping future spent Ni–Mo–V catalyst recycling, from regulations to corporate strategy, influencing technology choices, investments, and integration.

Table 12. Future drivers shaping spent catalyst recycling. Synthesized from Amato et al. (2025), Aromaa-Stubb et al. (2025), Baritto et al. (2025).

Driver	Expected impact
Regulation	Accelerates adoption
Circular economy	Integration with refineries
Digitalization	Process robustness
ESG metrics	Investment prioritization

The drivers indicate that catalyst recycling innovation will not rely solely on metallurgical efficiency. Regulations are likely to boost adoption by restricting disposal options, and circular economy principles support deeper integration of recycling into refineries. Digitalization will improve process robustness and monitoring, while ESG metrics will favor routes with clear environmental and social benefits alongside economic gains.

Conclusion

This review shows that recovering and recycling spent Ni–Mo–V catalysts is now a strategic issue tied to resource security, environmental regulations, and refinery sustainability. The maturity of hydrometallurgical and hybrid pyro–hydro methods indicates that metal recovery is not the main obstacle. Instead, challenges involve process integration, sulfur management, and system optimization.

A common limitation in studies is the fragmented treatment of metals and sulfur. Metal recovery is often optimized in isolation, while sulfur is treated as an emission or waste, undermining environmental and economic resilience. Integrated routes for sulfide oxidation, H_2S control, and sulfur recovery are rare but important for refinery-compatible circular schemes.

Another critical issue is the persistent gap between laboratory performance and industrial implementation. High recoveries under controlled conditions often overlook feed variability, gas-handling constraints, residue stabilization, and costs. Many promising concepts remain at low to intermediate TRL, not because of chemical infeasibility, but because scale-up and plant constraints are inadequately addressed.

From a sustainability perspective, the review confirms that recycling routes can outperform primary mining in terms of carbon footprint and material efficiency. However, this advantage is conditional. Processes with high energy demand, excessive reagent consumption, or poorly managed off-gases risk offsetting their environmental gains. Lifecycle thinking, therefore, must be treated as a design constraint, rather than a post hoc assessment.

The review emphasizes that future progress depends more on integration, modularization, and control than on new chemistries. Digital monitoring, adaptive control, and integrated layouts can improve robustness and reduce emissions. Spent Ni–Mo–V catalysts should be regarded as complex secondary resources that require systems engineering, rather than as waste or urban ore.

Overall, the field is mature but strategically incomplete. Closing gaps—metal–sulfur integration, low-emission operations, and scalability—will determine whether spent catalyst recycling becomes standard practice or remains a separate recovery technology.

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Conflicts of Interest

The author declares that there are no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Ethical Approval

Not applicable. This article is a review based exclusively on previously published literature and does not involve human participants or animal subjects.

Availability of Data and Materials

No new data were generated or analyzed in this study. All data discussed are derived from previously published sources cited in the reference list.

Authors' Contributions

The author was solely responsible for the conceptualization, literature selection, critical analysis, interpretation, and writing of the manuscript.

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