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Organic Removal from Spent Ni–Mo–V Hydroprocessing Catalysts: Mechanisms, Treatment Routes, and Process Integration—A Critical Review

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Abstract

Spent Ni–Mo–V-bearing hydroprocessing catalysts from heavy-oil refineries commonly contain free petroleum, adsorbed heavy hydrocarbons, carbonaceous coke, and sulfur- and nitrogen-bearing compounds. These distinct contaminant fractions create operational and environmental constraints before metal recovery, yet published removal efficiencies often rely on incompatible definitions and analytical bases. This critical review evaluates physical separation, solvent and surfactant washing, thermal desorption, pyrolysis, controlled oxidation, and hybrid treatment as pretreatment routes for subsequent hydrometallurgical or pyrometallurgical recovery rather than catalytic regeneration. A feed-specific mechanism–technology matrix, common mass-balance indicators, and a process-integration framework are proposed. Evidence suggests that solvent cleaning can target mobile oil without fully eliminating refractory coke, whereas staged thermal processing can access more strongly retained fractions at the expense of energy demand, emissions control, and phase changes. A reported mass-loss percentage cannot be equated automatically with oil removal, and concentration-based metal enrichment cannot establish quantitative metal retention. Future comparative work should quantify oil, coke, total carbon, sulfur species, metal losses, solvent closure, energy demand, emissions, and downstream metallurgy on consistent feed bases. The most defensible industrial choice is a staged, contaminant-specific pretreatment selected against explicit feed-quality requirements, rather than universal maximization of organic removal.

Keywords:

Spent hydroprocessing catalysts; NiMoV; petroleum-derived organics; solvent washing; thermal pretreatment; metal recovery

Highlights

- Oil removal, coke conversion, and desulfurization must be evaluated separately.
- Comparable performance requires constituent-specific mass and metal balances.
- Solvent washing and staged thermal processing have different operating trade-offs.
- The optimum cleaning endpoint depends on downstream metal recovery requirements.

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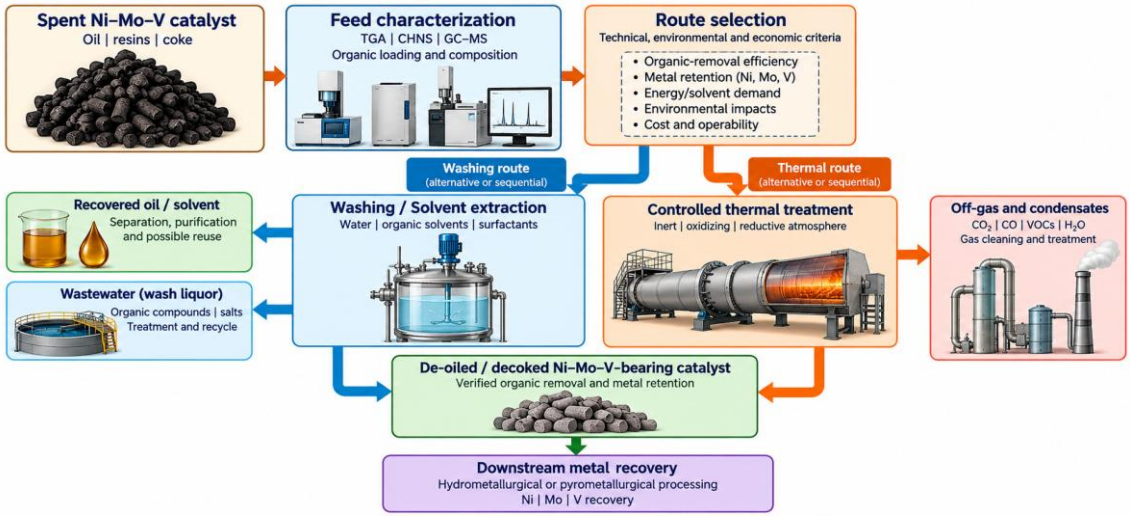
Graphical Abstract



Original schematic prepared for this review; arrows denote potential material pathways rather than validated industrial performance.

1. Introduction

1.1. Industrial context and scope

Hydroprocessing of heavy petroleum streams progressively deactivates supported Ni–Mo catalysts through the deposition of carbonaceous matter and metals, including vanadium. In spent catalyst lots, deposited V does not necessarily reflect an original catalytic component; it can come from the processed oil and coexist with Ni and Mo active-phase residues (Jurado & Ancheyta, 2022; Yu et al., 2025; Zhao et al., 2024). The resulting feed is heterogeneous across reactor bed layers, catalyst geometries, and storage history. This review addresses its preparation for metal recovery, not restoration of catalytic activity, which has fundamentally different acceptance criteria (Ahn et al., 2023; Klimov et al., 2022).

The central distinction is that free oil, adsorbed heavy organics, coke, and heteroatom-containing residues cannot be represented by a single “organic fraction.” Figure 1 illustrates their contrasting spatial accessibility, while Table 1 defines the associated analytical and process implications. Oil-bearing pellets may cause liquid carryover and increase solvent demand, but coke can remain after apparently successful solvent cleaning, changing heat release during roasting and leachant access (Esla et al., 2024; Hong et al., 2025; Madej et al., 2025).

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For orientation, Figure 2 outlines the relevant transport mechanisms; Figure 3 presents selectable treatment modules; Figure 4 sets the system boundary for the conservation equations; and Figure 5 defines an evidence-based decision sequence. Tables 2–7 supply analytical requirements, cross-route comparisons, reporting metrics, environmental safeguards, and research priorities. These figures and tables are conceptual synthesis tools and should not be interpreted as industrially validated equipment designs.

1.2. Critical question and contribution

The relevant question is not which treatment achieves the largest apparent loss of solid mass, but which contaminant-specific sequence delivers a qualified feed to Ni, Mo, and V recovery while controlling metal redistribution, solvent demand, emissions, and cost. Recent reviews address oily catalyst management, pretreatment, and metal-recovery flowsheets (Chen et al., 2025; Zhu et al., 2025; Yu et al., 2025; Gan et al., 2026). In contrast, the present review treats organic conditioning as an independently auditable engineering operation: removal from the solid is distinguished from product recovery; carbon, sulfur, solvent, and individual metal inventories are reconciled; and pretreatment acceptance is determined by matched downstream recovery experiments rather than visual cleanliness or concentration enrichment. This narrower focus avoids claiming novelty for technologies already covered comprehensively elsewhere.

2. Review Methodology and Evidence Assessment

2.1. Search scope and documentary status

The documentary register accompanying this manuscript contains 118 reference entries. Sources dated 2020–2026 were prioritized, while earlier studies were retained where they explained mechanisms, material properties, or process constraints. Search concepts included combinations of 'spent hydroprocessing catalyst' OR 'spent HDS catalyst' with 'oil removal' OR 'deoiling' OR 'solvent washing' OR 'coke oxidation' OR 'pyrolysis', and with 'nickel', 'molybdenum', 'vanadium', 'mass balance', or 'metal recovery'. The inherited compilation does not provide an independently verifiable database export, final search date, language restriction, deduplication log, or citation-chaining record; these cannot responsibly be reconstructed as retrospective search statistics. Accordingly, the work is a structured critical narrative review

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rather than a PRISMA-compliant systematic review or quantitative meta-analysis. Bibliographic metadata and DOI accuracy should be independently checked before submission.

2.2. Evidence classes and comparability

Evidence was interpreted within three author-developed classes: (i) direct experiments reporting a measured organic-removal endpoint on spent hydroprocessing catalysts, with the catalyst type explicitly specified; (ii) coupled studies measuring both pretreatment and downstream metal recovery; and (iii) adjacent studies on regeneration, FCC catalysts, general solvent chemistry, environmental methods, or related metallurgical systems. These classes are an author-developed interpretive framework, not a validated evidence-grading instrument. Evidence was not considered direct merely because it involved spent catalysts: an oil, coke, or another defined contaminant endpoint had to be measured. Reported results were compared quantitatively only when feed identity, analyte definition, mass basis, treatment conditions, and product boundaries could be reconciled.

2.3. Avoiding false equivalence

Changes in total carbon, total sample mass, solvent-extractable oil, or thermogravimetric loss are different observables. Likewise, greater Ni, Mo, or V concentrations after organic removal may arise from a reduced solid denominator even when no net metal recovery occurs. This review therefore separates measured removal from calculated enrichment and demands explicit elemental inventory closure wherever practical.

Cross-study interpretation first distinguishes NiMo, CoMo, Ni–Mo–V-bearing heavy-oil residue catalysts, other HDS catalysts, and FCC catalysts; the latter provide contextual rather than automatically transferable evidence. Similarly, regeneration and metal-leaching investigations inform potential downstream responses but cannot establish direct organic-removal performance unless a corresponding contaminant assay is reported (Ahn et al., 2023; Akhtar et al., 2024; Kohli et al., 2020; Latoof & Hassan, 2023; Quintanilha et al., 2022; Shafiq et al., 2022).

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These classification rules limit extrapolation from a title or process category to an engineering claim. Studies of different catalyst families are retained where they illuminate mechanisms, yet their measured endpoints and resulting uncertainty must remain visible rather than being merged into an artificial pooled removal percentage.

3. Characteristics of Petroleum-Derived Organic Contaminants

3.1. Four operationally distinct fractions

Residual free oil resides on external surfaces and accessible voids; capillary-held liquid accumulates in pore entrances and intrapellet networks. Resins and asphaltene-rich deposits can display stronger surface associations and broader molecular-weight distributions. Coke is a comparatively nonvolatile, insoluble carbonaceous deposit requiring oxidation or chemical transformation rather than simple dissolution. Sulfur- and nitrogen-bearing organics may distribute across extractable liquids and less accessible deposits and require emissions-aware treatment (Jurado & Ancheyta, 2022; Ovalles et al., 2017; Shakor et al., 2024).

Figure 1 provides an original spatial conceptualization of the contaminant classes and emphasizes why the order of operations matters. A freely drained pellet may still carry meaningful pore oil and coke, and a low total organic measurement need not imply metal-bearing pores are fully accessible.

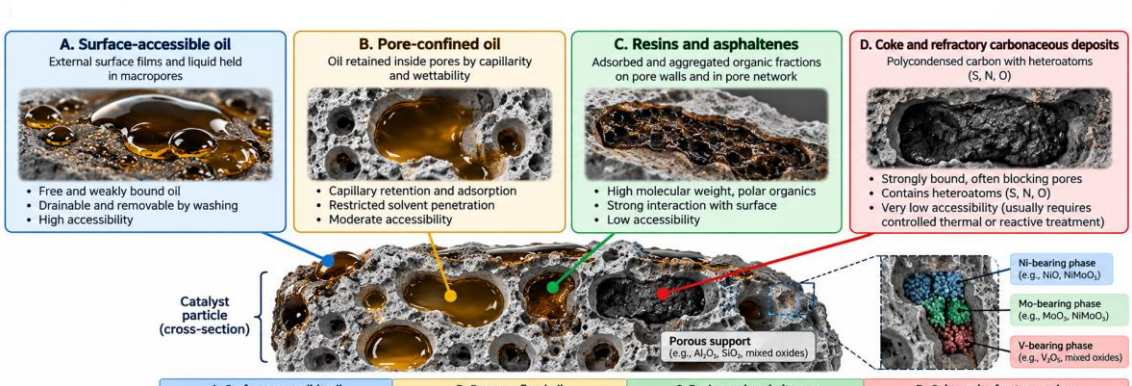

Figure 1. Organic contaminant populations in spent NiMoV hydroprocessing catalysts.
Note. Original conceptual synthesis informed by Jurado and Ancheyta (2022), Hong et al. (2025), and Shakor et al. (2024); not reproduced from a third-party figure.

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Each class can constrain downstream processing differently: liquid hydrocarbon affects handling and feed metering, heavier adsorbates can slow wetting, and coke can alter thermal ignition behavior and lixiviant access. Operational classification must therefore be based on analytical evidence, not appearance alone.

Table 1. Contaminant classes, likely location, and implications

Fraction	Likely state / site	Removal logic	Key risk
Free oil	Interparticle and outer surface	Drainage; solvent wash	Liquid carryover
Pore-retained oil	Accessible pores; capillaries	Warm wash; mass transfer	Incomplete extraction
Resins/asphaltenes	Strongly adsorbed layers	Solvent screening; hybrid	Slow desorption
Coke	Carbonaceous film/pore plugs	Controlled oxidation/pyrolysis	Heat release; gas emissions
S/N organics	Oil and solid deposits	Stage-specific treatment	SOx/NOx / odor

Note. Classification is qualitative and feed-dependent; adapted conceptually from Hong et al. (2025), Esla et al. (2024), and Chen et al. (2025).

3.2. Analytical triangulation

Thermogravimetric analysis and differential scanning calorimetry (TGA–DSC) identify mass-loss intervals and thermal events but cannot by themselves assign every event to petroleum evaporation or coke oxidation. CHNS elemental analysis constrains carbon, hydrogen, nitrogen, and sulfur, while chromatographic and spectroscopic techniques are needed to recognize extractable hydrocarbon families. Gas chromatography–mass spectrometry (GC–MS) of extraction liquors and Fourier-transform infrared spectroscopy (FTIR) of solids or extracts complement porosity, microscopy, and mineralogical characterization (Esla et al., 2024; Madej et al., 2025; Tian et al., 2024).

Table 2. Analytical methods, measured observables, and limits

Method	Primary observable	Interpretive caution
TGA–DSC	Mass loss/heat flow vs temperature	Multiple reactions may overlap
CHNS	Bulk elemental C, H, N, S	Carbon is not automatically extractable oil
GC–MS/extraction	Extractable organics fingerprint	Dependent on solvent and detection window
FTIR	Functional groups	Semi-quantitative without calibration

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Method	Primary observable	Interpretive caution
BET/porosimetry	Surface area / accessible porosity	Drying or heating changes texture
SEM–EDS / XRD	Morphology / inorganic phase change	Not a direct oil-mass assay

Note. Original comparative synthesis informed by Esla et al. (2024), Tian et al. (2024), and Bai et al. (2026).

A defensible data package uses at least a free/extractable-oil assay, a carbon balance, a sulfur assay, and a traceable drying protocol. This separation allows us to distinguish genuine hydrocarbon recovery from water release, gasification, and oxidation of carbonaceous solids.

Catalyst provenance and contact history change the interpretation of residual carbon: oil chemistry, deposition profiles, pore plugging, and metal loading can vary within a single lot. Accordingly, stratified sampling and independent extraction, CHNS, mineralogical, and thermal assays are preferable to a single bulk carbon figure (Ahmed et al., 2022; Chen et al., 2022a; Ebrahimi et al., 2023; Eleeza et al., 2021a, 2021b; Feng et al., 2024).

Heavy-feed chemistry is a second source of variability. Studies addressing residue and pore-scale behavior support explicit reporting of catalyst origin, prior service, oil accessibility, and treatment history rather than treating solvent affinity as a universal material constant (Fu et al., 2026; Jurado & Ancheyta, 2022; Kwao et al., 2026; Morales-Leal et al., 2023, 2024; Nguyen et al., 2021).

Thermal and mineralogical context further constrains the interpretation of coke-associated carbon. When comparing analytical datasets, sample preparation, retained moisture, oxidation atmosphere, and the denominator used for reported fractions must be identified (Novaes et al., 2022; Pathak et al., 2025; Pham et al., 2024; Shakor et al., 2024; Teng et al., 2023; Wang et al., 2020).

The most transferable research output is therefore a contaminant-resolved feed ledger with uncertainty and defined analytical endpoints, rather than an undifferentiated oil-and-coke estimate (Xu et al., 2025; Zhang et al., 2020; Zhu et al., 2022).

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4. Physicochemical Mechanisms Governing Organic Removal

4.1. Solvation, wetting, and accessible pores

Solvent cleaning initiates with contact and wetting, followed by dissolution or mobilization of oil and transport through a tortuous pore network. Solvent polarity alone cannot predict performance on multicomponent petroleum residues: affinity for light oil and affinity for polar heavy fractions may differ. The solvent must also be compatible with the inorganic catalyst surface, recovery unit, and subsequent leach chemistry (Abbas & Jung, 2024; Adavodi et al., 2025; Gao et al., 2021).

4.2. Transport resistance and kinetic endpoints

External-film renewal, intraparticle diffusion, capillary retention, and desorption may successively control removal. Increasing agitation reduces external-film resistance but cannot necessarily empty blocked pore networks. Mild heating can reduce liquid viscosity and speed transfer, whereas substantial heating can volatilize components and introduce a new emissions-control problem. Assess apparent extraction equilibrium using time-resolved oil inventories rather than a single terminal carbon assay (Gao et al., 2022; Hong et al., 2025).

These mechanisms are mapped in Figure 2. They imply that a negative laboratory result for a solvent may result from particle-scale accessibility as well as intrinsic solvency; conversely, grinding that accelerates cleaning may introduce fines and metal-bearing losses.

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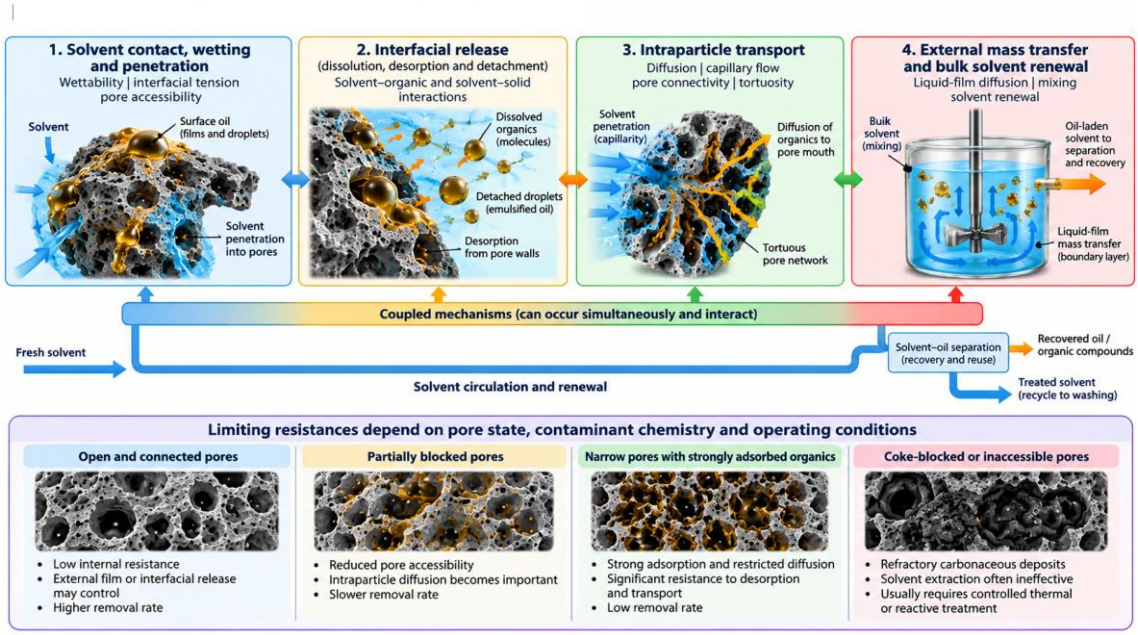

Figure 2. Physicochemical mechanisms and limiting resistances in organic removal.
Note. Original mechanism map synthesized from Abbas and Jung (2024), Gao et al. (2022), and Tian et al. (2024); not copied from the cited publications.

The framework supports staged experiments: measure external removal first, then assess pore diffusion with particle-size and residence-time variations, and finally determine whether remaining carbon responds to thermal treatment. The controlling regime must be established experimentally rather than assigned from a generic solvent property.

5. Mechanical and Physical Deoiling Technologies

5.1. Gravity, screening, and centrifugal separation

Initial gravity drainage, screening, and mechanical liquid separation can reduce immediately accessible petroleum without reagent addition. These operations are best understood as feed stabilization rather than comprehensive cleaning. Their effectiveness depends on liquid viscosity, pellet geometry, particle-size distribution, and free-liquid fraction. Their principal virtue is lower subsequent solvent or thermal duty; the main limitation is negligible removal of consolidated coke and adsorbed deposits (Feng et al., 2022; Hong et al., 2025).

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5.2. Mild thermal and vacuum-assisted conditioning

Warm conditioning, vacuum treatment, and controlled drying can decrease retained volatiles or support evaporation, but their apparent simplicity obscures vapor capture, ignition control, and indirect heat requirements. Vacuum does not guarantee removal of high-boiling fractions. The correct endpoint is a measured residual-oil specification combined with an acceptable off-gas envelope, not temperature alone.

Table 3 contrasts physical, solvent, and thermal pretreatment on compatible qualitative grounds. Quantitative removal rankings are intentionally omitted when literature measurements do not share a common analyte and feed basis.

Table 3. Function-based comparison of principal pretreatment methods

Method	Best-targeted fraction	Main advantage	Dominant limitation
Drainage/separation	Free liquid oil	Low reagent demand	Little pore or coke removal
Centrifugation	Mobilized interparticle oil	Rapid conditioning	Mechanical breakage / fines
Solvent washing	Extractable surface/pore oil	Recoverable liquid fraction	Solvent inventory / residual solvent
Surfactant + ultrasound	Interfacially trapped oil	Enhanced release	Emulsions / scale-up
Pyrolysis	Thermally decomposable organics	Condensable products possible	Energy / gas cleanup
Controlled roasting	Coke and sulfur-bearing solids	Deep decoking possible	Exotherms / phase changes

Note. Qualitative original synthesis adapted from Feng et al. (2022), Adavodi et al. (2025), Chen et al. (2025), and Tian et al. (2024).

6. Solvent-Based and Surfactant-Assisted Cleaning

6.1. Organic solvent classes

Nonpolar solvents such as hexane directly release petroleum fractions, as Madej et al. (2025) investigated for Ni–Mo–V-containing spent petrochemical catalysts. Polar solvents, including acetone, may remove a different mixture of extractables; Adavodi et al. (2025) specifically investigated acetone washing before V/Mo recovery without thermal pretreatment. These findings demonstrate feasibility for their tested materials but do not establish a universal solvent ranking across refinery feeds.

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Aromatic solvents may improve the dissolution of certain heavier hydrocarbon fractions, but selection should never rest on apparent removal alone. The comparison should consider flash point, toxicity, solvent recovery yield, solid/liquid ratio, extraction temperature, residual solvent in the product, and the compatibility of recovered oil with reuse options. Studies using surfactant/solvent mixtures indicate that interfacial engineering can improve removal in selected feeds, although it then creates a second aqueous-waste-management boundary (Gao et al., 2021; Gao et al., 2022).

6.2. Multistage countercurrent design

In a countercurrent washer, the cleanest solvent contacts the cleanest solid, improving the driving force near the discharge end. Nevertheless, a claimed advantage must be demonstrated on the total fresh-solvent makeup basis, including solvent remaining with wet solids, entrainment in removed oil, purges, and losses to vapor systems. The solvent recovery percentage and the pollutant removal percentage are independent quantities and must not be interchanged (Adavodi et al., 2025; D'Adamo et al., 2023).

6.3. Assisted aqueous cleaning

Ultrasound-assisted surfactant washing uses cavitation and local interfacial disruption to help mobilize oil. Tian et al. (2024) explored the mechanism of enhanced oil removal in spent hydrodesulfurization catalysts. The gains must be tested under industrially meaningful solids loadings: laboratory acoustic energy density and amplitude are not transferable merely by preserving the nominal irradiation time. Emulsion stability, wash-water recycling, and ultrasound equipment wear form part of the complete treatment cost.

Table 4 summarizes the strongest applicable evidence without converting unlike outcomes into a single efficiency ranking.

Table 4. Selected direct organic-removal studies and evidence limitations

Study	Primary route	Reported outcome type	Comparability warning
Madej et al. (2025)	Hexane extraction	Oil fraction removed (>40% in tested conditions)	Oil-specific basis
Adavodi et al. (2025)	Acetone washing	Elemental C/H/S change; high solvent recovery	Carbon is not oil mass

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Study	Primary route	Reported outcome type	Comparability warning
Gao et al. (2021).	Solvent/surfactant mixture	Crude-oil cleaning response	Feed- and formulation-specific
Tian et al. (2024).	Ultrasound/surfactant	Enhanced oil-removal mechanism	Acoustic scale dependence
Chen et al. (2025).	Pyrolysis + roasting	Distinct deoiling/decoking/desulfurization	Includes chemical mass transformations

Note. Adapted as an original evidence classification from the cited studies. Numeric outcomes are not pooled because denominators and analytes differ. The 2025 two-stage study reports 22.4 wt% mass loss attributed to deoiling under its specified experimental conditions.

Solvent results should be interpreted as demonstrations for particular catalyst and solvent combinations, not transferable solvent rankings. The solvent–solid mass balance must also distinguish petroleum recovered as product from hydrocarbons retained in distillation bottoms and residues (Abbas & Jung, 2024a; Adavodi et al., 2025; Feng et al., 2022; Gao et al., 2021, 2022b; Hong et al., 2025).

For industrial selection, the governing trade-off is extractable-oil removal versus fresh solvent makeup, product contamination, and residual coke. Hexane and related solvent studies therefore inform route screening without proving complete cleaning of heavier deposits (Madej et al., 2025; Ovalles et al., 2017; Yang et al., 2016).

7. Thermal and Thermochemical Organic Removal

7.1. Thermal desorption and pyrolysis

Thermal treatments divide into drying or desorption of volatile fractions, pyrolysis of organic layers under limited oxygen, and oxidative conversion of coke-rich residues. These operations can interact: pyrolysis changes the remaining coke composition and creates gas or condensables, whereas subsequent oxidation consumes carbon, releases heat, and can modify sulfide phases. Gao et al. (2023) reported an oil-recovery-oriented catalytic pyrolysis concept, while Chen et al. (2025) assessed a sequential pyrolysis–blank-roasting strategy for oily spent hydroprocessing catalysts.

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7.2. Controlled oxidation and solid phase changes

Rapid oxygen access to carbon-rich solids can induce local exotherms and heterogeneous reaction fronts. Control requires gas composition, temperature gradients, residence-time distribution, and evolving heat-release measurements—not simply furnace set-point control. A kinetic model for coke and sulfur forms underscores their coupled oxidation behavior (Abdulla et al., 2025). Meanwhile, oxidative treatment can alter the crystallinity and leachability of Ni, Mo, and V-bearing phases, so decoking effectiveness must be judged alongside downstream recovery response (Bai et al., 2026; Yu et al., 2024).

7.3. Two-stage pretreatment and apparent efficiency

Chen et al. (2025) reported a laboratory two-stage sequence with a 42.2 wt% overall mass reduction under their specified conditions, subdivided into a 22.4 wt% deoiling contribution and a further 19.8 wt% associated with desulfurization and decoking. The authors reported very high decoking and desulfurization degrees for this feed and test design. These component-specific percentages should not be generalized to spent NiMoV catalysts with different oil histories, coke reactivity, and metal deposition. In particular, losing sulfur as gaseous products may be metallurgically advantageous but imposes gas-cleaning responsibilities.

Table 5 contrasts thermal stages and reports why duty, gaseous products, and phase transformation must accompany removal claims.

Table 5. Thermal pretreatment stages and control requirements

Stage	Dominant transformation	Required monitoring	Downstream concern
Drying / desorption	Evaporation of liquids	Vapor mass, LEL, condensate	Residual organic fractions
Pyrolysis	Thermal cleavage/volatilization	Heating rate, gas composition, oil yield	Residual char chemistry
Oxidative decoking	Carbon combustion; sulfur conversion	O_2, CO/CO_2, SOx, hot spots	Metal phase transformation
Sequential treatment	Partitioning of volatile and refractory fractions	Interstage mass and energy closure	Integrated emissions and residues

Note. Adapted conceptually from Gao et al. (2023), Chen et al. (2025), Abdulla et al. (2025), and Bai et al. (2026).

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Thermal pathways must be interpreted as coupled carbon, sulfur, oxygen, and mineral-phase transformations. Oxidative regeneration, roasting for leaching, and direct deoiling are different experimental objectives and should not be pooled as one treatment efficacy.

The oxidation literature describes different objectives and therefore different endpoints: removal of combustible deposits, regeneration of catalytic activity, and formation of phases suitable for metal extraction are not interchangeable. Coupled thermal histories may alter leachability and sulfur release; reporting must connect the inferred reaction path to solid and off-gas observations (Abdulla et al., 2025; Arslanoğlu & Yaraş, 2021; Bai et al., 2026; Bian et al., 2025; Chen et al., 2025a, 2026).

Atmosphere, oxygen transfer, and reactor temperature profiles condition whether carbon converts to CO, CO_2, condensable compounds, or residual char. Related studies establish mechanistic possibilities, not a common quantitative benchmark across heterogeneous feeds (Chen et al., 2024; Cui et al., 2023; Esla et al., 2024; Fang et al., 2024; Frydl & Vu, 2026; Gao et al., 2022a).

Pyrolysis and oxidative decoking should consequently be evaluated as separate modules, with measured liquid yield, gas speciation, reaction heat, and the characterization of retained mineral phases. Adjacent regeneration studies provide useful design warnings but do not replace direct tests on Ni–Mo–V-bearing feeds (Gao et al., 2023; Klimov et al., 2022; Li et al., 2020; Na et al., 2024; Pham et al., 2025; Shi et al., 2024).

For oxidation trials, report oxygen demand, gas-phase carbon and sulfur, bed temperature gradients, residence-time distribution, and changes to Ni/Mo/V-bearing phases. These measurements distinguish deep decoking from an uncontrolled material transformation (Singh et al., 2025; Srour et al., 2019, 2021a, 2021b; Vogt et al., 2023; Wang et al., 2021a).

Pilot transfer cannot be inferred solely from setpoint temperature or total mass loss; the required evidence also includes hot-spot management, off-gas treatment, and matched downstream extraction (Wang et al., 2026; Yu et al., 2024a).

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8. Emerging and Hybrid Cleaning Technologies

8.1. Ultrasound and other intensification

Mechanical–solvent combinations and ultrasound can intensify liquid release through disruption of stagnant boundary layers and improved wetting. Direct published evidence for ultrasound-assisted surfactant cleaning exists in spent HDS catalysts (Tian et al., 2024). For microwave, supercritical-fluid, and pressurized-solvent concepts, promising performance on other solid matrices does not prove equivalent removal or commercial readiness for oily NiMoV catalysts. Any adoption proposal must document penetration depth, pressure integrity, solvent interactions, feed variability, and complete process mass balances.

8.2. Hybrid-sequence logic

The strongest process rationale for hybrids is functional rather than technological novelty. Physical draining first recovers free oil; solvent washing targets soluble and accessible organics; a later oxygen-controlled stage addresses coke if required by the metallurgical route. However, each added step increases utility, containment, and operating complexity. Figure 3 illustrates these optional modules and explicitly avoids presenting them as a universally optimal sequence (Feng et al., 2022; Adavodi et al., 2025; Chen et al., 2025).

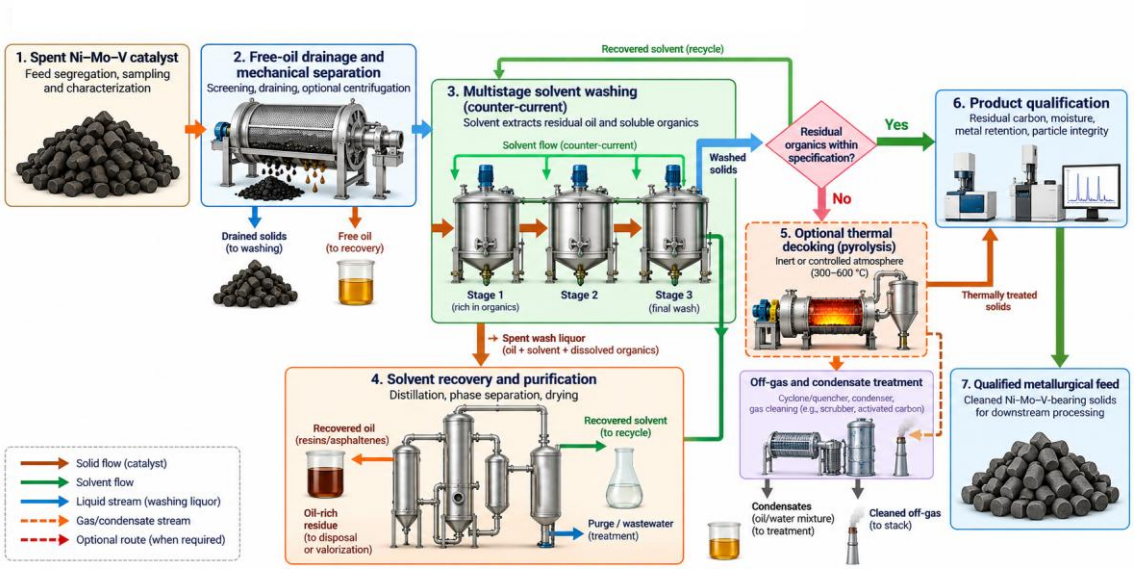

Figure 3. Modular treatment pathways for organic conditioning before metal recovery.

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Note. Original block-flow synthesis adapted from Feng et al. (2022), Adavodi et al. (2025), and Chen et al. (2025). Solvent recovery and thermal treatment are optional branches, not an established plant design.

The preferred arrangement depends on whether the downstream unit tolerates a specified residual carbon content, requires low liquid hydrocarbons for safe handling, or benefits from prior sulfide oxidation. Demonstrating one route on intact catalyst particles does not establish its feasibility for fines-rich or agglomerated spent material.

9. Process Integration and Mass–Energy Balances

9.1. A common material-accounting basis

A traceable inventory begins with a stated as-received or dry catalyst basis, including moisture, extractable oil, coke-like carbon, sulfur, inorganic solids, and individual Ni, Mo, and V masses. Removal from treated solids must be distinguished from recovery as a useful output: for constituent i, $\eta_{i,solid}$ (%) = 100 × [$m_{i,feed}$ − $m_{i,treated\ solid}$]/$m_{i,feed}$; for recovered petroleum, R_{oil} (%) = 100 × $m_{oil,recovered\ product}/m_{oil,feed}$. Carbon disappearance from the treated solid is expressed as X_c (%) = 100 × [$m_{c,feed}$ − $m_{c,residual\ solid}$]/$m_{c,feed}$, whereas closure is C_c (%) = 100 × $\Sigma m_{c,measured\ output\ streams}/m_{c,feed}$. An analogous sulfur closure includes solid, liquid, condensate, wastewater, and gas streams. The feed and outlet carbon contributed by any solvent or auxiliary reagent must be kept separately or included consistently in a full elemental balance.

For each M ∈ {Ni, Mo, V}, report retained metal $R_{m,solid}$ (%) = 100 × $m_{m,treated\ solid}/m_{m,feed}$; nonproduct allocation $L_{m,nonproduct}$ (%) = 100 × $\Sigma m_{m,nonproduct\ streams}/m_{m,feed}$; and analytical closure C_m (%) = 100 × $\Sigma m_{m,all\ measured\ outputs}/m_{m,feed}$. Captured fines, wash liquor, filter cake, and aerosols must remain inside the boundary; unmeasured balance residual is not evidence of physical loss. Define solvent recovery R_s (%) = 100 × $m_{s,recovered}/m_{s,sent\ to\ recovery}$ and makeup intensity I_s = $m_{s,fresh\ makeup}/m_{catalyst,feed}$. Separately record solvent entrained with wet solids or recovered oil, vapor losses, purge, and residual solvent in the conditioned catalyst. Use purchased energy per

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tonne of original catalyst, including electricity, imported heat, solvent distillation, auxiliary systems, and thermal startup. Product oil cannot be credited both as saleable recovered fuel and as displaced process energy without an explicit boundary.

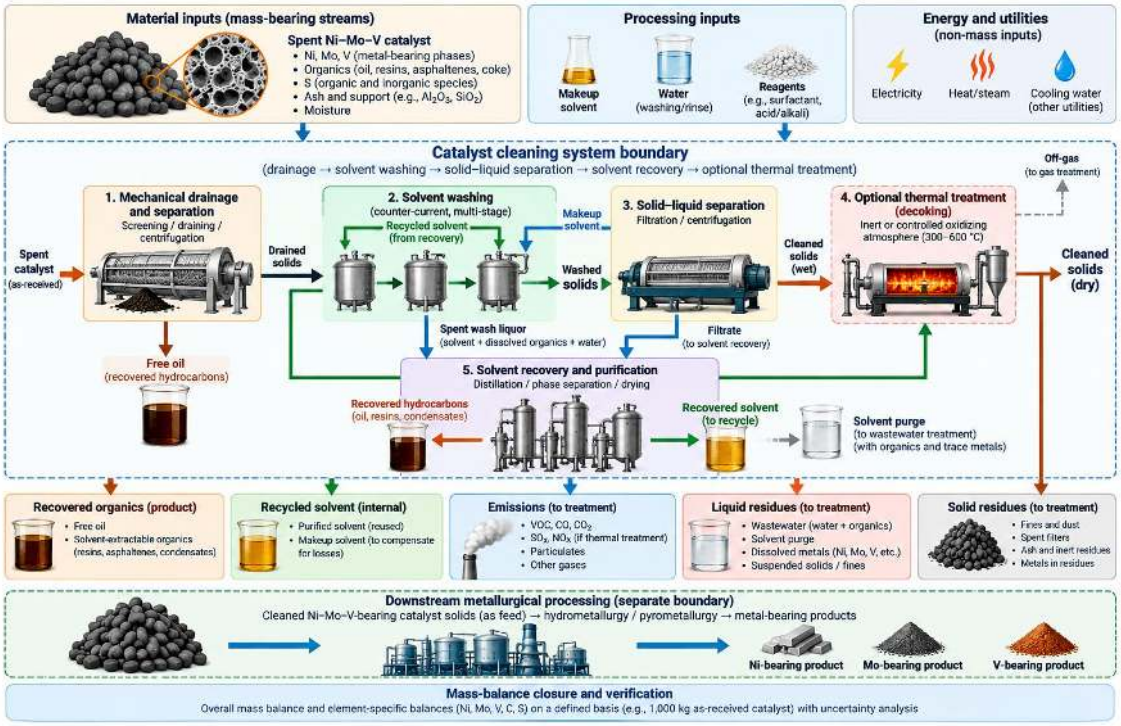

Figure 4. Integrated mass-balance boundary for catalyst pretreatment and downstream metallurgy. *Note.* Original balance schematic; relevant integration questions are adapted from D'Adamo et al. (2023), Le and Lee (2021), and Yu et al. (2025).

Capturing liquid organic and solvent outlets separately makes it possible to determine both their recovery value and the net losses. Thermal routes additionally require off-gas carbon and sulfur accounting; reduced solid mass must not automatically be assigned to recovered petroleum.

For thermal processes, record condensable petroleum, permanent gas, CO/CO_2, sulfur-bearing emissions, char, and residual solid separately. An apparent carbon-conversion value alone cannot serve as closure. Gas volumetric flows require temperature, pressure, wet/dry basis, and calibration; sulfur species include SO_x and H_2S where chemically relevant. A complete route-to-product indicator is $R_{m,overall}$ (%) = 100 × $m_{m,final\ qualified\ product}/m_{m,original\ spent\ catalyst}$, calculated independently for Ni, Mo, and V. This metric should complement, not substitute for, pretreatment-stage metal retention.

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9.2. Energy integration and metal recovery

Solvent heating and distillation can dominate wash-route thermal duty, while pyrolysis and oxidation demand start-up energy and controlled management of volatile- and coke-derived heating values. Waste-heat recovery is feasible only after reconciling operating profiles, off-gas contamination, and heat-exchanger fouling risks. Downstream oxide-forming roasting and leaching may exploit pretreatment phase changes, but they can also increase impurity dissolution or create refractory products (Arslanoğlu & Yaraş, 2021; Bai et al., 2026; Fang et al., 2024).

Table 6. Minimum normalized indicators for comparing cleaning routes

Metric	Definition / measurement	Required reporting unit
Oil-removal efficiency	Extractable oil removed / initial extractable oil	% of initial oil
Coke conversion	Coke-like C removed / initial coke-like C	% initial coke C
Metal retention	Elemental Ni, Mo, V in treated solid / feed inventory	% for each metal
Fresh-solvent intensity	Solvent makeup / as-received feed	kg/t catalyst
Solvent recovery	Solvent returned / solvent sent to recovery	% of defined cycle
Purchased energy	Electricity + net imported heat, defined boundary	MJ/t catalyst
Solid product quality	Residual oil, C, S, PSD, moisture	wt%; particle sizes
Off-gas and waste	Stream-resolved gas, aqueous, and solids	kg/t catalyst

Note. Proposed reporting standard developed in this review; measurements require a common dry or as-received mass basis and closure uncertainty.

A downstream comparison must finally test leaching kinetics or pyrometallurgical operation on both treated and untreated representative feeds. Otherwise, a cleaner-looking catalyst is only an intermediate process achievement, not a verified enhancement of Ni–Mo–V extraction (Cai et al., 2022; Liang et al., 2022).

Downstream metallurgy sets the practical acceptance limit for cleaned solids. The following process literature illuminates the potential consequences of residual organics and altered mineral phases but does not alone prove a particular pretreatment is optimal.

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Pretreatment-related metal redistribution may occur through soluble species, fines, and thermal phase changes. For hydrometallurgical comparisons, matched untreated controls should establish whether the new feed actually changes leaching rates, reagent requirements, filterability, and product purity; studies on solvent-enabled and selective metallurgical recovery provide route context, not a common pretreatment benchmark (Abbas & Jung, 2024b; Cai et al., 2022, 2025; Chen et al., 2022b, 2023; Gan et al., 2026).

Evidence on product separation and associated flowsheet operations identifies potential acceptance limits for washed or decoked catalysts, particularly changes in solution chemistry and impurity behavior (Gao et al., 2024; Gomes et al., 2024; Han et al., 2023; Huang et al., 2022; Ilhan et al., 2020; Lee et al., 2026).

For both leaching and smelting routes, gross metal enrichment can reflect removal of oil and water from the denominator. A defensible recovery gain therefore requires individual element inventories from original feed through final saleable product (Liang et al., 2022; Liu et al., 2025; Lv et al., 2023; Padh et al., 2019; Phann et al., 2022; Pradhan et al., 2020a).

Choice of the hydrometallurgical route can change the useful degree of pretreatment. Sulfur and carbon tolerances, alkali consumption, filtration resistance, and phase dissolution should be measured rather than assumed invariant (Pradhan et al., 2020b; Shi et al., 2021, 2023; Srivastava et al., 2025; Taz et al., 2024; Tian et al., 2026).

Industrial acceptance further depends on product specification and the management of residues and circulating liquors. The comparison must encompass downstream purification and reject streams rather than isolate the pretreatment unit (Türk & Arslanoğlu, 2025; Wang et al., 2021b; Wen et al., 2023; Wu et al., 2021; Yaraş & Arslanoğlu, 2020; Ying et al., 2026).

This product-directed perspective is consistent with the wider Ni, Mo, and V recovery literature, but the magnitude of any pretreatment benefit remains feed- and route-specific (Ying et al., 2025; Yu et al., 2025; Zhao et al., 2024b; Zhu et al., 2025).

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10. Environmental, Safety, and Techno-Economic Assessment

10.1. Risk distribution rather than risk elimination

Solvent washing transfers hydrocarbons to a liquid recovery circuit and introduces flammable-liquid storage, pump sealing, and vapor-control requirements. Surfactants may produce contaminated aqueous effluents and stable emulsions. Thermal routes avoid large circulating solvent inventories but can emit volatile organic compounds (VOCs), CO, CO_2, sulfur oxides, and particulate matter; they also require ignition and self-heating controls. The relevant question is where each hazard is transferred within the integrated facility (Chen et al., 2025; Chen et al., 2025; Tian et al., 2024).

10.2. Economics and life-cycle boundaries

Capital expenditure must include not only primary contactors or furnaces but also inerting, fire protection, condensers, solvent purification, off-gas treatment, residue collection, and monitoring systems. Operating expenditure must reflect solvent make-up, utilities, maintenance, wastewater treatment, and labor. Net value should credit only marketable recovered hydrocarbon fractions, avoided waste-handling costs, and measurable increases in metal recovery, while avoiding double counting (D'Adamo et al., 2023; Aromaa-Stubb et al., 2025).

Comparisons of environmental impacts must impose the same functional unit and system boundary, including downstream treatment where it materially changes because of pretreatment. Life-cycle assessment (LCA) of catalyst-metal recovery can inform accounting categories, but it should not be mistaken for a direct comparison of oil-removal pretreatments (Aromaa-Stubb et al., 2025).

Table 7. Safety, environmental, and economic gates for route screening

Criterion	Solvent route	Thermal route
Main hazard	Flammable vapors, solvent loss	Hot spots, off-gas, combustion
Pollution-control burden	VOC control; spent wash liquor	Gas cleaning; particulates; SOx
Major utility	Pumping + solvent regeneration	Heating + induced draft / cooling
Key economic sensitivity	Solvent consumption and reuse	Energy and off-gas treatment

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Criterion	Solvent route	Thermal route
Necessary demonstration	Closed-cycle solvent and residue balance	Stable hot-spot and emissions management

Note. Original comparative assessment informed by Chen et al. (2025), D'Adamo et al. (2023), and Aromaa-Stubb et al. (2025).

Environmental comparisons must account for the whole system, including solvent losses, wash liquors, off-gas treatment, solid residues, utilities, and metals recovered. Adjacent circular-economy studies are informative for boundary selection, not automatically comparable life-cycle inventories.

Economic and environmental interpretation needs one boundary encompassing oil recovery, solvent recycling, utility supply, emissions abatement, and metals ultimately delivered. Environmental research supplies accounting principles and selected route evidence rather than transferable pretreatment life-cycle values (Aromaa-Stubb et al., 2025; Chen et al., 2025b; D'Adamo et al., 2023; Le & Lee, 2021; Marafi et al., 2024; Pathak et al., 2022).

Reported benefits should be conditioned on feed loading, avoided disposal, solvent attrition, and verified downstream metal yield. Broader recovery assessments support such a system boundary but do not automatically validate an individual cleaning technology (Pereira et al., 2026a; Yu et al., 2024b).

Evidence from comprehensive reviews of catalyst management and metal recycling establishes the broader technology landscape (Chen et al., 2025; Zhu et al., 2025; Yu et al., 2025; Gan et al., 2026). The specific contribution of the present work is a contaminant-resolved pretreatment acceptance framework rather than a new claim to an exclusive technology route: analytical identity, constituent mass closure, solvent and purchased-energy ledgers, metal disposition, and controlled downstream acceptance tests are treated as jointly necessary. This comparison prevents broad reviews of recovery technology from being mistaken for quantitative evidence of the best organic-removal endpoint.

11. Critical Comparison, Industrial Readiness, and Research Gaps

11.1. Evidence quality and process maturity

Laboratory demonstration should be interpreted against tested catalyst provenance and solids throughput. Pilot studies of the overall metal recovery sequence, including those

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reviewed by Yu et al. (2025), provide valuable engineering context but do not prove that any particular organic cleaning step has commercial qualification. Technology readiness level (TRL) labels are useful only when a defined integrated unit has corresponding operating evidence, so numerical TRL assignments are deliberately withheld where the available records do not support them.

11.2. Decision synthesis

Figure 5 translates the critical comparison into sequential decisions: diagnose contaminant classes; screen routes for targeted removal; quantify element-specific retention; evaluate safety, utilities, secondary emissions, and final metallurgical response; then test the preferred option under representative continuous or cyclic conditions. An attractive percentage removal from a small batch is insufficient without closure on feed variability and solvent or off-gas recycle.

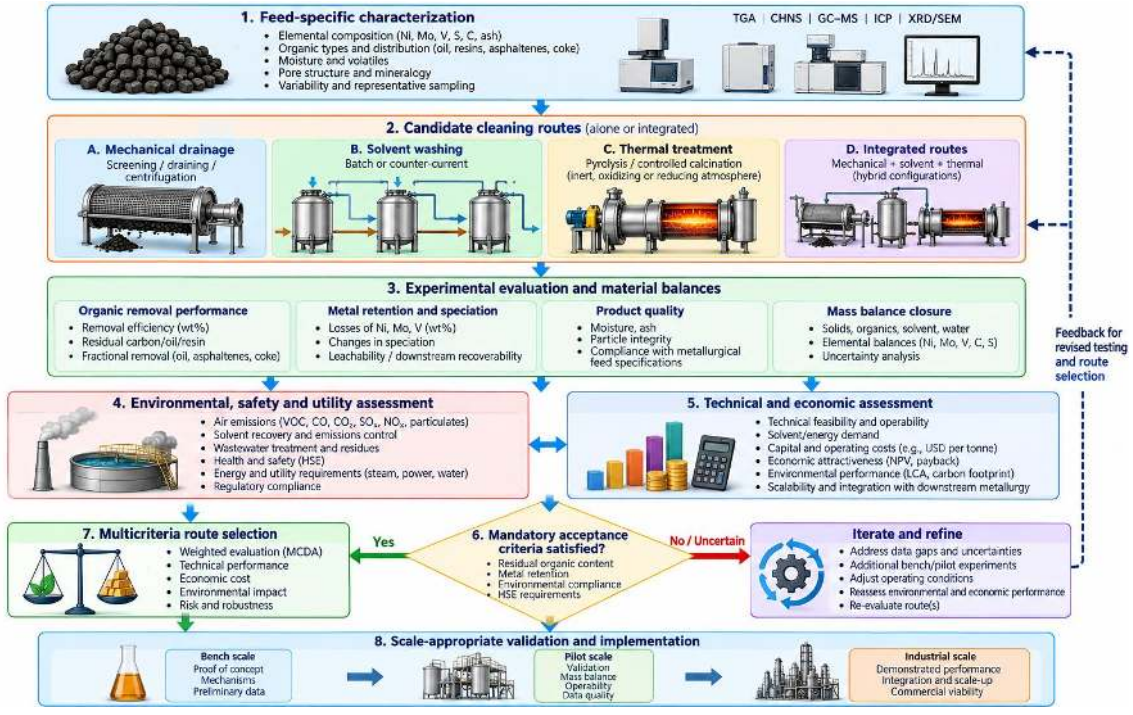

Figure 5. Evidence-based technology selection and scale-up assessment.
Note. Original decision framework synthesized from Adavodi et al. (2025), Chen et al. (2025), Tian et al. (2024), and D'Adamo et al. (2023).

Major data gaps remain in multicomponent organic speciation, separation of free oil and coke removal, element-by-element metal loss to fines and liquors, long-run solvent deterioration, simultaneous gas-phase sulfur and carbon balances, and comparative tests of

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downstream leachability or smelting response. Reporting standards should prioritize uncertainty intervals, representative feed sampling, and transparent mass-closure criteria (Le & Lee, 2021; Liang et al., 2022; Zhao et al., 2024).

11.3. Verification program

A practical verification sequence begins with composite sampling across lot heterogeneity and replicate TGA–DSC, CHNS, solvent-extractable oil, PSD, and elemental assays. Bench screening should compare physical drainage, at least two solvent chemistries, ultrasound-assisted washing where feasible, and staged thermal treatment. Each test should quantify product yield and Ni/Mo/V inventories. The best two routes should proceed to integrated trials with solvent recycle or controlled off-gas capture and matched downstream metallurgical assays; residual uncertainty, rather than nominal laboratory yield, should govern advancement.

Commercial transferability requires attention to equipment-specific hydrodynamics, reaction kinetics, physical integrity, and representative feed variability. The following sources offer neighboring engineering approaches and are cited as transferability or research-priority context.

For scale-up, feed representativeness and physical integrity are indispensable. Neighboring process studies can guide experimental design, residence-time measurement, acoustic energy accounting, and equipment selection, but must not be treated as proof of industrial treatment capacity for oily NiMoV catalysts (Adavodi et al., 2026; Alsuwaidi et al., 2025; Huang et al., 2020; Li et al., 2016; Ma et al., 2025; Pereira et al., 2026b).

Pilot trials should register fresh solvent makeup and solvent entrainment, as well as actual acoustic energy intensity (kWh/t catalyst) for ultrasound routes or purchased fuel and electricity for thermal routes. Continuous operation introduces recycle degradation, fouling, solids attrition, and gas/aqueous discharge constraints absent from small batches (Pinto & Soares, 2013; Tian et al., 2024; Wang et al., 2018; Xhaferaj & Ferella, 2022; Xu et al., 2026; Yang et al., 2018).

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A final qualification campaign should compare replicates of untreated and treated feed under identical downstream recovery conditions, with uncertainty bounds on element-specific mass closure and final metal product yield (Zhao et al., 2024a; Zoraga et al., 2024).

12. Conclusions and Future Perspectives

Petroleum-derived contamination of spent NiMoV hydroprocessing catalysts consists of fundamentally different free, adsorbed, and carbonized fractions that require different removal mechanisms. No single laboratory removal percentage represents overall pretreatment success. Physical separation stabilizes oil-rich feed, solvent processes selectively remove accessible extractable fractions, and controlled thermal treatment can address refractory carbon and sulfur-containing deposits but may change inorganic phases and emission burdens. A rational flowsheet is staged and contingent on the downstream Ni–Mo–V recovery operation's acceptance requirements. Universal solvent rankings, generic TRL values, and claims of complete carbon removal are not justified without comparable feed analyses and validated balances. Progress toward industrial implementation requires constituent-specific removal data, verified metal retention, closed solvent and off-gas inventories, measured net energy use, safety assessment, and matched downstream recovery demonstrations.

Declarations

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Author contributions: Conceptualization, literature review, critical synthesis, and manuscript preparation: Antonio Clareti Pereira.

Data availability: No new experimental datasets were generated. The bibliographic register and evidence classification were derived from the supplied literature inventory.

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