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Energy Balance Closure and Thermophysical Property Uncertainty in Spent NiMo and NiMoV Catalyst Recovery: Heat Effects, Thermal Integration, and Predictive Reliability—A Critical Review

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Abstract

Spent nickel–molybdenum (NiMo) and nickel–molybdenum–vanadium (NiMoV) hydroprocessing catalysts combine valuable metals with oils, coke, sulfides, and complex oxide supports. Their recovery routes integrate thermal pretreatment, oxidative or alkaline roasting, aqueous separation, and product finishing, but energy claims are often incomparable because boundaries, reference states, thermophysical assumptions, and unmeasured heat losses differ. This critical review develops a property-consistent framework for steady-state and transient energy-balance closure, evaluates sensible and latent heat and reaction enthalpy contributions, and connects thermal integration to uncertainty-aware route assessment. The review critically evaluates a predominantly 2020–2026 bibliography supplemented with a directly relevant route model and foundational reconciliation literature; evidence is evaluated by relevance and transferability rather than assumed uniform quality. The proposed framework distinguishes metered utilities, calculated stream enthalpies, reaction heat, and residual losses; specifies minimum reporting data for solids, gases, and aqueous streams; and integrates reconciliation, sensitivity analysis, and uncertainty propagation. The principal conclusion is that credible energy comparisons require closed elemental and phase inventories, consistent enthalpy reference states, and explicit uncertainty intervals before heat recovery or route superiority can be claimed.

Keywords: Spent hydroprocessing catalysts; Energy balance closure; Thermophysical uncertainty; NiMoV recovery; Heat integration; Data reconciliation

Highlights

- Energy closure requires explicit stream, reaction, utility, and loss accounting.
- Thermophysical uncertainty can reverse apparent route-energy rankings.
- Heat recovery is constrained by temperature, fouling, and gas chemistry.
- A traceable reconciliation workflow improves predictive reliability.

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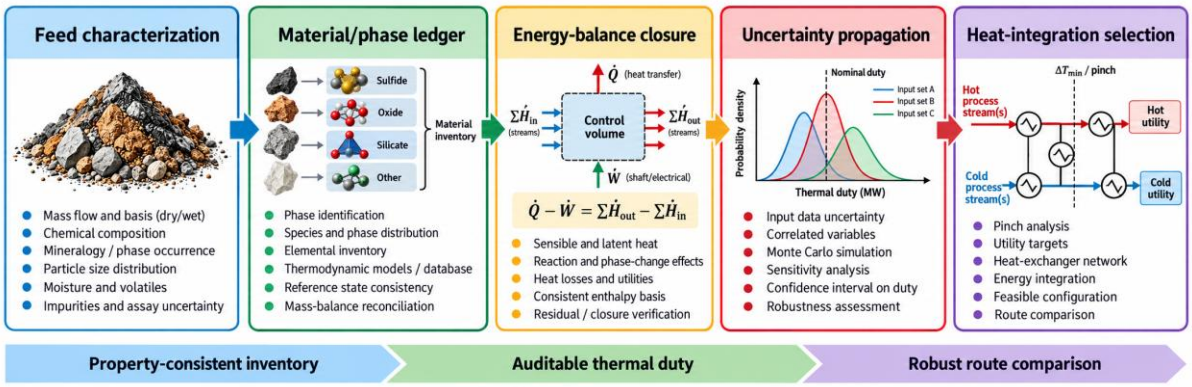
Graphical abstract



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1. Introduction

Spent hydroprocessing catalysts are both a strategic secondary source of molybdenum, nickel, and, in certain services, vanadium, and a chemically heterogeneous thermal processing challenge. Coke and oil content, sulfur, support composition, metal deportment, and intended products govern the processing route. Reviews and comparative recovery studies establish substantial technical diversity but do not provide interchangeable energy intensities because thermal inventories and system boundaries vary (Amato et al., 2025; Le et al., 2021; Liang et al., 2022; Gan et al., 2026).

This review shifts emphasis from nominal heating temperature to an auditable thermodynamic ledger. Figure 1 establishes the unit-operation boundary; Figure 2 presents the data-quality and closure logic; Figure 3 develops the heat-integration cascade; Figure 4 specifies uncertainty propagation; and Figure 5 distinguishes useful energy from exhaust, losses, and unexplained residuals. Table 1 identifies the governing quality thresholds, Table 2 defines property requirements, Table 3 separates duty terms by operation, Table 4 frames

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reaction-enthalpy evidence, and Table 5 provides a decision-oriented reporting checklist. The graphical abstract summarizes the integrated argument.

Unlike the recent technology-centered reviews by Chen et al. (2025), Dong et al. (2025), Ying et al. (2025), and Gan et al. (2026), the contribution here is the combined accounting architecture: harmonized control volumes, enthalpy reference states, property provenance, measurable closure residuals, data reconciliation, and uncertainty-informed comparison. Route-specific mass-and-energy models already exist: Baritto et al. (2022) modeled salt roasting, water leaching, and product recovery for vanadium from bitumen-upgrading spent catalyst. The remaining gap is not the absence of any model, but the lack of transferable, property-consistent and uncertainty-aware cross-route comparisons.

The critical question is not merely whether route A operates at a lower furnace set point than route B. The critical question is whether both routes are compared using the same wet-feed basis, energy-carrier equivalence, physical-property functions, chemical-reaction basis, and useful-product specification. Without those requirements, apparent efficiency gains may be attributable to accounting asymmetry rather than intrinsic process performance.

2. Review Methodology and Thermal Data Quality

2.1 Scope, documentary basis, and limitations

The documentary basis comprises 107 entries in the submitted reference list, mainly dated 2020–2026. The additional directly relevant process-model study of Baritto et al. (2022) and selected foundational methodological sources are included in the revised bibliography. Sources were interpreted as direct spent-catalyst evidence, adjacent process evidence, or methodological transfer. This is a structured critical narrative review, not a systematic review or meta-analysis. Neither an exhaustive database export nor independently retrievable screening logs were supplied with the initial manuscript; consequently, a reproducible PRISMA search flow or a universal DOI-verification claim is not made. Citation metadata should be checked against publisher records during final production.

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Evidence is classified as direct (spent hydroprocessing NiMo/NiMoV experimental work), adjacent (other spent catalysts or related metallurgical transformations), or methodological (pinch, reconciliation, optimization, or life-cycle inventory). A paper's inclusion does not imply that it contains direct thermal-duty measurements. A high-confidence route comparison requires comparable boundary definitions, feed assay, heat and electricity metering, phase accounting, error bounds, and repeatability.

For a reproducible update, a proposed search protocol is: Google Scholar, Scopus, and Web of Science; publication window 2020–2026 for process applications, with earlier foundational sources retained when methodologically indispensable; English-language scholarly articles and books; queries combining ("spent catalyst" OR "hydroprocessing catalyst" OR "NiMoV") AND ("energy balance" OR "heat duty" OR "roasting" OR "thermophysical" OR "process simulation"), and ("data reconciliation" OR "gross error detection" OR "pinch analysis" OR "exergy" OR "uncertainty propagation"). Records should be de-duplicated by DOI and title and assessed for process relevance, boundaries, phase specification, measured versus modeled duties, and uncertainty disclosure. Backward/forward citation chaining is recommended. This is a proposed, transparent update procedure—not a claim that these databases, steps, or a specific final search date were used in the original corpus.

Table 1 turns these requirements into an operational hierarchy. No numerical acceptance threshold is imposed as universal: a permissible residual must reflect the instrument uncertainty and the intended engineering decision.

The four evidence levels in Table 1 constitute an author-developed descriptive hierarchy, not an externally validated grading instrument. In particular, a route model that estimates heat duties does not establish that its utilities were independently metered or that its residual passed a statistical closure test.

Table 1. Evidence quality and closure requirements for thermal process assessment

Evidence level	Minimum reportable content	Critical interpretation

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Direct measured	Feed/flow rates, temperature, utilities, compositions	Suitable for duty estimation if instrument uncertainty is known
Reconciled direct	Measured streams plus covariance and conservation constraints	Suitable for comparative closure when residual tests pass
Calculated indirect	Phase balance, $C_p(T)$, reaction and latent heat estimates	Dependent on property model and composition uncertainty
Transferred method	Industrial pinch, optimization, or LCA framework	Useful analytically; not evidence of NiMoV plant performance

Note. Original classification developed in this review; methodological principles are informed by the cited reconciliation and process-analysis literature.

This classification prevents a frequently overlooked category error: a thermodynamic equilibrium prediction and a measured industrial heat demand may both be valid but answer different questions.

3. Energy Balance Boundaries and Reference States

3.1 Control volumes and closure equation

For a control volume, neglecting kinetic and potential energy only when justified, the first law is $dU/dt = \dot{Q} - \dot{W} + \Sigma in(\dot{m}h) - \Sigma out(\dot{m}h)$. Heat transfer $\dot{Q}$ is defined positive into the volume and shaft work $\dot{W}$ positive outward. Enthalpy must use a consistent chemical reference convention: either species enthalpies include formation contributions or explicit reaction-enthalpy terms are added, but never both for the same transformation. Material inventories include all feed, product, recycle, dust, exhaust, condensate, and liquid-loss streams.

At steady state, define the absolute energy residual $R_E = \dot{Q} + \Sigma in(\dot{m}h) - \dot{W} - \Sigma out(\dot{m}h)$. Its normalized magnitude is $\varepsilon_E = 100|R_E|/E_throughput$ (%), where $E_throughput = |\dot{Q}| + |\dot{W}| + \Sigma in|\dot{m}h| + \Sigma out|\dot{m}h|$, evaluated under the declared reference state and with the same convention across compared routes. This denominator is a diagnostic scale, not a thermodynamic efficiency or a universally standardized index. Report absolute and normalized residuals with propagated uncertainty; do not prescribe a universal allowable percent. A

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nonzero R_E may reflect actual unmetered transfer or unmodeled/biased data, and must never automatically be labeled insulation heat loss.

Before interpreting R_E, test total mass and element closure, at minimum Ni, Mo, V, Al, S, C, water, and Na for alkaline routes. Carbon can exit as coke, oil, CO, CO_2, hydrocarbons, or condensates; sulfur can remain in sulfides/sulfates or leave as SO_2, SO_3, H_2S, dissolved sulfate, or condensate. Report raw and reconciled flow measurements independently. Reference conditions of 298.15 K and 1 atm are a useful default for formation-enthalpy calculations, but actual choices, reference phases and water state (liquid, vapor, adsorbed or bound) must be declared rather than assumed.

Figure 1 maps practical ledger boundaries for combined thermal and aqueous routes. The figure intentionally avoids assumed heating efficiencies and does not equate reaction yield with energy efficiency.

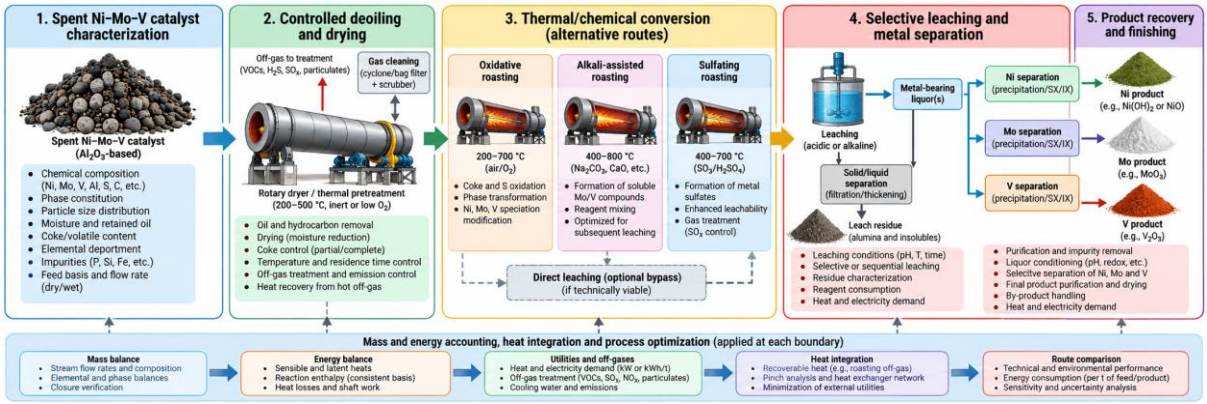

Figure 1. Control-volume boundaries across spent NiMo(V) catalyst recovery.
Note. Original figure prepared for this review by the author with AI-assisted visualization; not reproduced or adapted from a specific published image.

The unit-operation decomposition enables local closure before reconciling the plant-wide energy ledger. Recycle-loop temperatures and moisture inventories must be kept consistent at all boundaries.

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3.2 Functional units and accounting basis

Comparisons should report at least MJ per tonne of as-received spent catalyst, MJ per tonne of dry catalyst, and where appropriate MJ per kilogram of saleable recovered metal or product. Each basis answers a different question; high-yield operations cannot be preferred energetically without reconciling differing product quality, residue quantities, and utility demands. The chosen reference temperature, pressure, phases, and water state must be declared.

Report purchased fuel (with heating-value basis), electricity, steam (supply state and condensate return), compressed air, fan power, agitation, pumping, filtration, cooling, solvent regeneration, evaporators, drying, calcination, and effluent treatment separately before any justified aggregation. A product-normalized comparison must state recovered metal yield, product purity, hydration state and saleable product specification (e.g., MoO_3, V_2O_5, or $NiSO_4 \cdot 6H_2O$).

4. Heat Capacities and Thermophysical Property Data

Spent catalysts are mixtures of alumina or other supports, sulfides, oxides, coke, volatile organics, absorbed liquid, and conversion products. A mass-weighted mixture heat capacity can provide an initial estimate only when phase fractions and temperature-dependent component functions are available. Reactions, devolatilization, and pore moisture invalidate the assumption that one constant effective heat capacity can represent every heating interval.

A reproducible property ledger records, for each phase and component, the source/database or measurement method, $C_p(T)$ correlation and validity interval, density, latent heat, formation enthalpy, assumed phase fractions, uncertainty and extrapolations. Extrapolation of polynomial heat capacities beyond validated ranges should be flagged and sensitivity-tested. DSC/TGA with off-gas analysis can check thermal-event assignments but must not be represented as a full-scale furnace-duty measurement.

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Table 2 states the minimum property ledger. Source-specific data, where available, should supersede simple mixture approximations; measured calorimetry is preferable for heterogeneous, reactive materials.

Table 2. Thermophysical input data and principal sources of model uncertainty

Material/stream	Property needed	Major uncertainty driver
Oily wet catalyst	$C_p(T)$, moisture, vaporization heat	Bound water, oil composition, coke loading
Roaster solids	$C_p(T)$, phase composition, reaction extent	Transformation kinetics, support composition
Off-gas	Species composition, $C_p(T)$, flow rate	Excess air, sulfur species, moisture
Liquors and slurries	Density, $C_p(T)$, mixing and dissolution enthalpy	Dissolved salts, solids fraction
Final products	$C_p(T)$, crystallisation/phase transitions	Hydration state, purity and phase changes

Note. The listed properties are required model inputs rather than numerical literature averages; units should be standardized before calculation.

For a stream heated without phase change, $\Delta H_{sens} = m\int C_p(T)dT$. For an effective multiphase solid, $\Sigma_i m_i\int C_{p,i}(T)dT$ is preferable to a single C_p assumption if phase fractions are known. For temperature intervals containing decomposition, melting, vaporization, or solid-state conversion, latent and reaction terms must be added consistently.

The sensible enthalpy change is evaluated over the actual inlet and outlet temperatures. When the mixture is reactive or undergoes a phase change, a single averaged heat capacity cannot replace the required enthalpy-path calculation; standard formation enthalpies and reaction-heat terms must be combined using one convention only.

5. Sensible and Latent Heat Contributions

5.1 Dry solids, bound liquid, and evaporation

The sensible-duty approximation is often adequate for preliminary screening when material composition remains stable, and temperatures are moderate. However, wet or oily spent catalysts consume energy through liquid heating and phase changes; with incomplete moisture analysis, uncertainty in latent duty can be as important as uncertainty in the dry-solid

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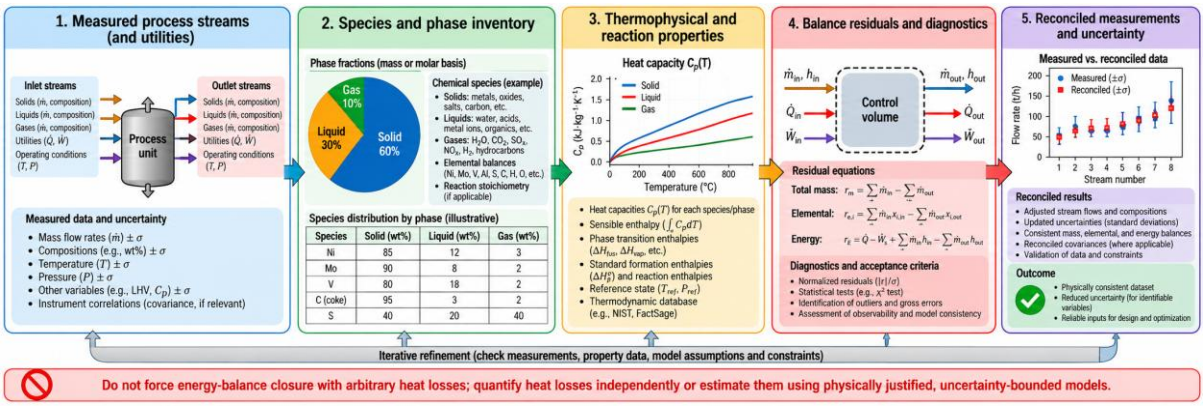
heat capacity. Dry-feed normalization conceals that penalty unless original moisture content remains visible.

Free surface or pore water, bound or crystallization water, retained petroleum liquids and coke require separate mass fractions and thermodynamic paths. A dry-basis headline must be accompanied by as-received moisture, oil and coke inventories. When saleable oil provides a real displacement credit, a transparent upper accounting form is Q_credit = m_oil × LHV_oil × η_usable; η_usable includes demonstrated collection and useful conversion, and is zero where recovered fuel is not actually usable. Theoretical combustion heat from uncollected hydrocarbons or partially oxidized coke is not a credit.

5.2 No double counting across heating intervals

A complete enthalpy ledger assigns each phase transformation to one defined interval. Evaporation enthalpy should not be counted both as a stepwise latent term and through steam enthalpy in the outlet stream. Similarly, standard heats of formation and separately added reaction enthalpies must not be combined unless the accounting convention is explicitly reconciled.

Figure 2 presents a practical closure sequence in which properties and species inventories precede interpretation of residual error.



Species	Solid (wt%)	Liquid (wt%)	Gas (wt%)
Ni	85	12	3
Mo	90	8	2
V	80	18	2
C (coke)	95	3	2
S	40	20	40

Figure 2. Measurement–property–balance sequence for closure and reconciliation.

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A residual can indicate missing species, biased instruments, an incorrect phase assumption, or unmodeled heat transfer. It should be treated as a diagnostic rather than corrected by an arbitrary furnace-efficiency factor.

6. Reaction Enthalpies during Oxidation and Roasting

6.1 Sulfide oxidation, coke combustion, and alkali roasting

Oxidative conversion of metal sulfides, coke combustion, sulfate formation, alkali reactions, and phase rearrangements alter the net thermal duty. Temperature control may be exothermic over some intervals but simultaneously require significant preheating, water evaporation, and flue-gas sensible heat. Thermodynamic favorability alone does not establish extent of conversion or measured heat release; kinetics, residence time, oxygen potential, and particle transport must be evaluated.

Table 3 isolates the accounting terms to prevent route-to-route comparisons from overlooking downstream thermal operations.

Table 3. Operation-specific energy terms and comparability limitations

Operation	Include in duty ledger	Comparability pitfall
Drying/deoiling	Feed heating, evaporation, exhaust, recovered oils	Wet versus dry feed basis
Pyrolysis/decoking	Reaction energy, purge heating, gas cooling	Unmeasured combustibles and off-gas
Oxidative/soda roasting	Sensible and reaction heat, air, dust, exhaust	Conversion extent not measured
Leaching/extraction	Solution heating, agitation, pumping, cooling	Electrical duties omitted
Crystallisation/calcination	Evaporation, cooling, phase change, exhaust	Solvent/water recycles excluded

Note. Conceptual duty ledger for screening; quantitative values must be obtained by measurement or validated calculation.

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As a result, an efficient metal recovery pathway can still be thermally inferior if the process requires concentrated alkali make-up, high-temperature roasting, extensive wastewater evaporation, or repeated drying of intermediates.

6.2 Reaction heat evidence and reference-state control

Reaction enthalpies can be computed as $\Delta H^{\circ}_{rxn} = \Sigma \nu_p \Delta H^{\circ}_{f,p} - \Sigma \nu_r \Delta H^{\circ}_{f,r}$ at the same reference state. At operating temperature, a heat-capacity correction is required. The calculation is only defensible when chemical speciation, oxidation state, reaction extent, and transformed phases are explicitly documented.

A reaction contribution is evaluated from measured or justified reaction extent ξ: $Q_{rxn} = \xi \Delta H_{rxn}(T)$, using a declared sign convention and composition-specific heat-capacity correction. Neither complete coke combustion nor complete sulfide oxidation may be assumed without carbon/sulfur balances and $CO/CO_2/SO_x/H_2S$ and solid-phase evidence. For soda roasting, document reacted alkali and identified product phases; do not infer reaction heat solely from feed alkali dose.

For a defined reaction, the standard enthalpy of reaction requires signed stoichiometric coefficients and formation enthalpies at a common reference temperature and phase state. Its process contribution is proportional to the reaction extent; an assumed complete conversion is not a substitute for gas and solid speciation.

Table 4 provides the appropriate classification for reaction assumptions in a review without a common calorimetric dataset.

Table 4. Reaction-enthalpy claims and evidence necessary for quantitative use

Thermal event	Model requirement	Verification need
Sulfide oxidation	Sulfur/metal oxidation state and ΔH° formation	Roaster gas and solid assay
Coke combustion	Coke amount, extent, O_2 utilization	CO/CO_2/off-gas carbon balance
Soda-assisted conversion	Balanced stoichiometry and phase identity	Alkali consumption and product phase analysis

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Sulfate decomposition	Reaction direction vs. temperature and pO_2	Gas-phase sulfur monitoring
Hydration/dehydration	Water form and phase equilibrium	TGA/DSC and moisture analysis

Note. No universal enthalpy values are asserted because composition, phase constitution, and reaction extent vary across feeds.

This classification is especially important in literature where oxidation temperature and metal leaching yield are reported, but heat flux, off-gas composition, and reaction conversion are absent.

7. Thermal Duties in Drying, Pyrolysis, and Decoking

7.1 Oil and coke as variable energy carriers

Deoiling and pyrolysis can recover organics, reduce hazardous handling risks, and shift the oxidation burden downstream. Whether recovered organics constitute useful energy depends on their purity, heating value, combustion or saleability, and the treatment required for sulfur-bearing gases. Credit must never be assigned merely because a fraction is described as recoverable oil.

7.2 Thermal measurements and mass transfer

Thermogravimetric analysis, differential scanning calorimetry, gas analysis, and repeated feed assays can separate moisture loss, volatile release, carbon oxidation, and sulfate/oxide transitions. Laboratory ramps should not be translated directly to industrial residence time because convection, particle-size distribution, bed depth, and gas–solid contacting alter the effective thermal history.

8. Energy Requirements for Leaching and Product Recovery

8.1 Aqueous operations and their hidden energy penalties

Room-temperature extraction can avoid furnace duty but remains dependent on pumping, stirring, liquid–solid separation, reagent preparation, solution conditioning, and

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downstream concentration. Hot acidic or alkaline leaching requires both heating of the liquor inventory and associated losses. Concentrated solutions may exhibit nonideal heat capacity, viscosity, and dissolution enthalpy, so pure-water assumptions should be flagged as screening approximations.

8.2 Recovery yield versus delivered product specification

A fair comparison specifies the target metal purity, hydrated or anhydrous product form, fraction of unrecovered metals, volume of spent liquor, and the number of evaporation or calcination steps. Low-temperature separation routes can have substantial total energy demand if solvent recovery or product drying is excluded.

9. Heat Losses, Recovery, and Process Integration

9.1 Heat-source mapping and practicable integration

Theoretical exhaust enthalpy is not synonymous with usable recovered heat. Heat grade, contamination, corrosion, sulfur-bearing gases, particulate fouling, process intermittency, heat-exchanger approach temperature, and maintenance constraints determine accessible savings. A pinch-based analysis should be conducted on confirmed hot and cold streams, using measured or defensible design temperatures and heat capacities.

Any proposed network should identify hot- and cold-stream temperature intervals, target minimum approach temperature ΔT_{min}, temporal overlap, annual operating availability, bypass periods and cleaning downtime. A practical recovery estimate also deducts pressure-drop/fan and pump penalties, and respects sulfuric acid dew point, condensate handling, corrosion, dust/vanadium-bearing fouling and contaminated heat-exchanger surfaces. These requirements make gross exhaust enthalpy an upper opportunity bound, not a validated savings claim.

Figure 3 illustrates a cascade of potential heat users, from upstream air/feed preheating to lower-grade washing or solution duties. Its arrows describe opportunity, not proven viability.

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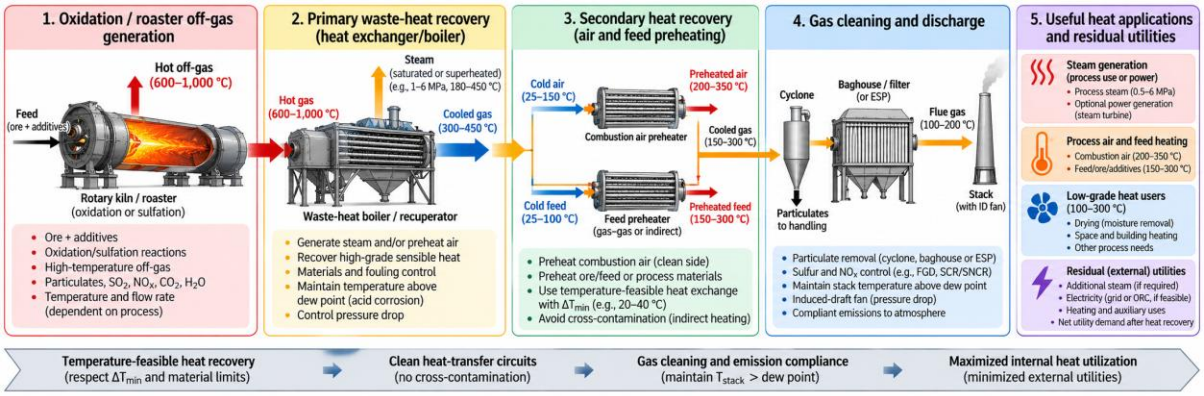

Figure 3. Conceptual heat integration cascade and screening constraints.
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In plants processing sulfur- and vanadium-bearing catalysts, acid dew point, dust deposition, and metal-bearing condensate may constrain downstream exchangers. This can sharply reduce economic recovery below the gross sensible-heat potential.

9.2 Interpreting first-law losses and exergy

First-law recovery potential is associated with energy quantity, whereas exergy also penalizes low-temperature energy quality. Therefore, a route that recovers a large quantity of low-grade heat cannot automatically displace an equivalent amount of high-temperature furnace energy. Electric heating and heat pumps should be compared using site-specific electricity assumptions and their feasible temperature lift.

For a nonreacting stream at reference environment (T_0,p_0), the specific physical exergy may be expressed as $ex_ph = (h - h_0) - T_0(s - s_0)$, neglecting kinetic/potential contributions. Chemical exergy, where material, fuel or reaction streams are involved, is distinct and requires consistent environmental and chemical reference conventions. Exergy diagnoses useful work potential and energy quality; it does not replace first-law closure or establish exchanger feasibility.

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10. Energy Balance Reconciliation, Property Uncertainty, and Research Gaps

A reliable process model integrates material-balance closure, phase-specific property functions, direct utility metering, and uncertainty-aware reconciliation. Figure 4 provides the sequence; Figure 5 shows where energy residuals enter the accounting.

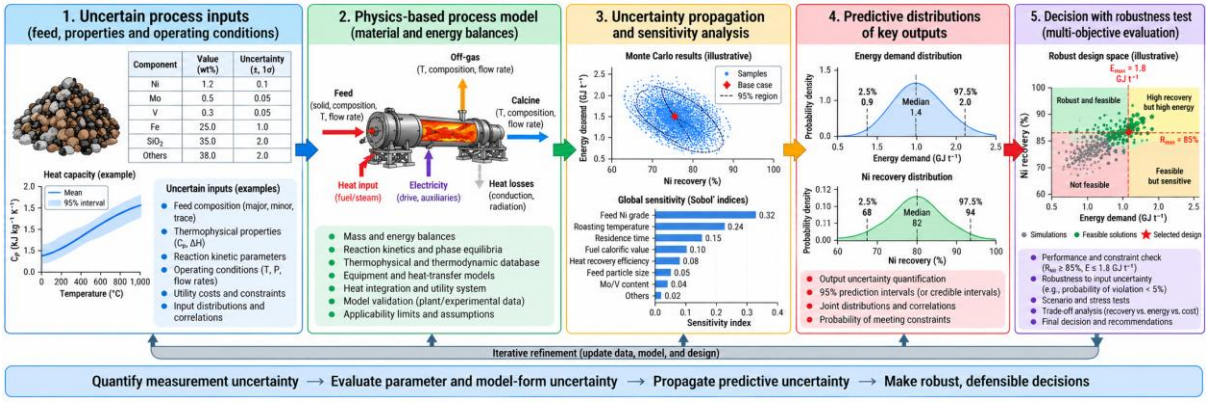

Figure 4. Uncertainty propagation and decision-reliability workflow.
Note. Original table developed for this review; the entries are reporting requirements and not measured performance data.

Local sensitivities may be estimated as $S_i = (x_i/Q)(\partial Q/\partial x_i)$, where Q is the selected energy metric and x_i is an uncertain input. A first-order uncertainty approximation is $Var(Q) \approx J\Sigma J^T$, with J the gradient of Q with respect to inputs and Σ the input covariance matrix. Monte Carlo propagation is preferable when nonlinear reactions, thresholds, correlated properties, or discrete phase changes render linear approximations unreliable.

For prospective comparison, specify uncertain input distributions and their empirical basis, separating feed heterogeneity (aleatory) from uncertain correlations, calibrations and structural assumptions (epistemic). Random meter errors, suspected systematic biases, and covariance should be handled separately. For Monte Carlo route comparisons, report random seed or reproducible sampling protocol, simulation count, convergence checks, interval estimates, and the pairwise probability $P(E_A < E_B)$; apparent ranking should be flagged as nonrobust when the uncertainty interval for the difference crosses zero. No route-ranking

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probabilities are calculated in this narrative review because harmonized route datasets were not available.

The covariance matrix must be expressed in units consistent with the input vector. When the output energy metric is close to zero, normalized local sensitivities can be unstable; absolute derivatives, scenario comparisons, and interval estimates should therefore also be reported.

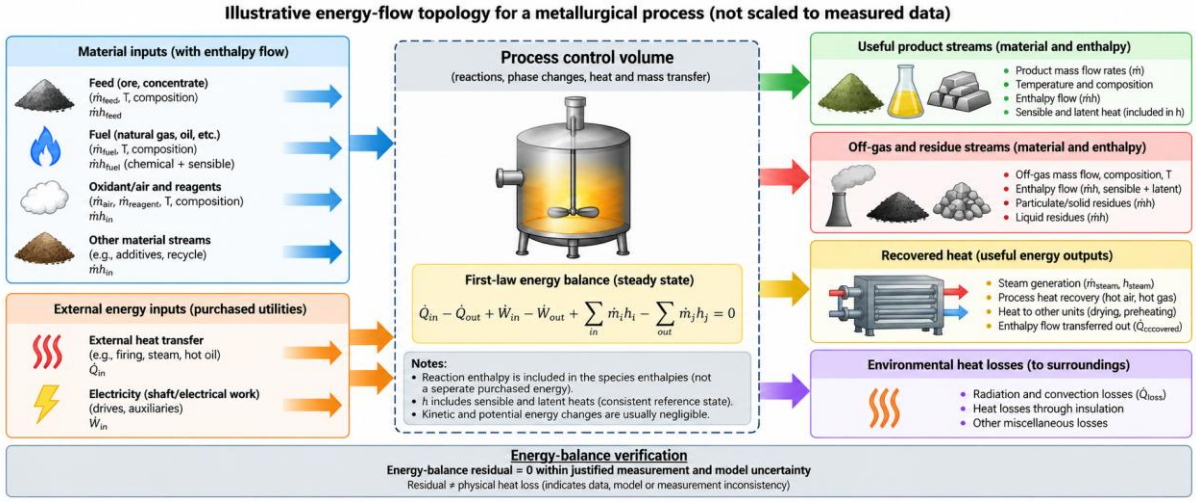

Figure 5. Energy ledger topology separating useful output, exhaust, losses, and unexplained residual. Note. Original figure prepared for this review by the author with AI-assisted visualization; not reproduced or adapted from a specific published image. Arrow widths do not indicate measured quantities.

Reconciliation should impose measured conservation constraints without hiding instrument defects. A statistically significant energy residual can be evidence of an incorrect species model, untracked hot recycle, wrong gas humidity, or systematic temperature bias. Reconciled data should be accompanied by original measurements, estimated covariance, constraints, residuals, and sensitivity to property correlations.

In a measured process, generalized least squares can reconcile process variables subject to mass/energy constraints, followed by residual and gross-error tests. A low adjusted residual is not sufficient evidence of instrument accuracy when systematic bias or model mismatch remains. Narasimhan and Jordache (1999) provide foundational treatment of

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reconciliation and gross-error detection; these methods are transferable but do not substitute for NiMoV-specific validation.

Table 5 compiles the minimum information needed for an auditable comparison. Rather than assigning generic technology readiness levels, the framework evaluates evidence strength, model scope, and confidence of the thermal comparison; technology readiness becomes relevant only when industrial demonstration status materially alters feasibility.

Table 5. Minimum data and reporting checklist for robust route-energy comparison

Dimension	Required evidence	Decision test
Feed inventory	Wet/dry flow, assay, oil/coke, sulfur, moisture	Same reference basis?
Streams and states	Temperatures, pressures, phases, gas/liq flow	All major outlets captured?
Properties	$C_p(T)$, phase changes, ΔH reactions, sources	Traceable data and intervals?
Utilities	Fuel, electricity, steam, air, cooling, efficiency	No omitted auxiliary duty?
Closure	Measured vs computed duty, residual, error bars	Residual consistent with uncertainty?
Integration	Heat-source/sink profiles, exchanger constraints	Recoverable not just theoretical heat?
Products and residues	Yield, purity, water duty, emissions	Equivalent product specification?

Note. Original table developed for this review; entries specify reporting requirements, not measured performance.

Three gaps dominate: scarcity of matched calorimetric and flow data for heterogeneous spent feeds, incomplete oxidation and exhaust speciation in thermal-route studies, and weak propagation of property uncertainties into process-level rankings. These gaps motivate representative feed sampling, phase-resolved measurements, meter calibration, independently reconciled balances, and pilot designs that capture utilities and contaminated heat-recovery limitations.

A defensible validation campaign should pair repeated representative feed assays with independently metered fuel, electricity, inlet and outlet stream rates, off-gas species, moisture, and product phase composition. The resulting independent closure residuals and uncertainty intervals should be made available before assigning a preferred route.

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10.1 Critical synthesis of available evidence

Direct spent-catalyst recovery and integrated route models establish feasible metal-extraction sequences, but the energy evidence is heterogeneous. Baritto et al. (2022), for instance, developed mass and energy balances for a bitumen-upgrading spent-catalyst V recovery flowsheet including salt roasting, water leaching and product generation, and used them for sizing and techno-economic evaluation. That route-specific modeled balance is more relevant than a generic technology analogy, but it cannot alone validate cross-route thermophysical data, loss estimation or confidence intervals. Recovery-focused reviews (Chen et al., 2025; Dong et al., 2025; Ying et al., 2025; Gan et al., 2026) provide process-context breadth without establishing a common independently metered NiMoV energy benchmark.

10.2 Aqueous and thermal route evidence

Leaching, ionic-liquid and selective-separation papers constrain recovery chemistry and product options; absent metered agitation, heating, solvent regeneration and product-finishing utilities, extraction efficiency is not an energy-intensity observation (Abbas & Jung, 2024a, 2024b; Arslanoğlu et al., 2021; Le et al., 2021). Thermal pretreatment and roasting studies contribute transformation temperatures and reaction pathways, but temperature programs and TGA/DSC mass losses are not equivalent to exhaust-, off-gas- and wall-loss-inclusive plant duties (Amato et al., 2025; Liang et al., 2022). Cross-industry heat-integration studies help screen potential exchanger networks (Boldyryev et al., 2025; Walmsley et al., 2024) but do not directly demonstrate NiMoV hot-gas equipment reliability.

10.3 Model validation, data quality, and remaining gaps

The evidentiary hierarchy should therefore distinguish (i) audited metered utilities, (ii) simulation- or stoichiometry-derived heat duties, (iii) thermophysical datasets and reaction evidence, (iv) process simulation and economic estimates, and (v) generic methodological analogs. Life-cycle emissions and techno-economic cost estimates answer different questions

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from calorimetry and first-law closure. The most pressing comparative gap is paired pilot measurements of feed species and moisture, meter-calibrated fuel/electricity, exhaust/condensate and hot-solid streams, reliable properties, tested closure residuals and propagated uncertainty on an equal product basis. No numerical process ranking or measured recovery credit is inferred from the present heterogeneous literature.

11. Conclusions

Meaningful comparison of energy performance in spent NiMo and NiMoV catalyst recovery requires a common material basis and consistent thermodynamic reference states. The proposed framework separates direct measurements, calculated stream enthalpies, reaction effects, and unexplained residuals. It thereby avoids treating missing energy as a freely adjustable heat-loss term.

Sensible and latent heat, chemical transformations, off-gas handling, and downstream aqueous/product finishing must be evaluated as one integrated system. Heat-integration options are constrained by temperature quality, contamination, corrosion, and feasible exchanger design. Reconciled balances with explicit uncertainty intervals provide a stronger foundation for scale-up decisions than isolated operating temperatures or reported metal recoveries.

The principal limitation is the lack of a homogeneous experimental thermal dataset across the reviewed routes. Priority research includes coupled calorimetry and chemical phase analysis, full gas and liquid inventories, uncertainty-aware model validation, and pilot-scale metering under comparable product specifications. These steps would enable predictive thermal benchmarks and more defensible technology selection.

Declarations

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Data availability. No original experimental datasets were generated for this critical narrative review. The evidence base consists of the references listed in the manuscript. No structured screening log or computational route-comparison dataset is provided; independent bibliographic and DOI checks remain part of the author's pre-submission quality control.

Author contributions. The author was responsible for conceptualization, methodology, literature synthesis, critical analysis, visualization, and manuscript preparation.

Use of AI-assisted tools. AI-assisted tools were used for drafting support, language editing, and conceptual figure visualization. The author remains responsible for the scientific interpretation, accuracy of statements, reference verification, and compliance of all figures with the target journal's editorial policy. This disclosure does not imply that independent bibliographic verification is complete.

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