

BOTTLE ROLL TESTS FOR KINETIC EVALUATION OF NICKEL ORES: CAPABILITIES, LIMITATIONS, AND MISINTERPRETATIONS – A CRITICAL REVIEW

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Abstract

Bottle roll testing is widely used to evaluate leaching performance due to its simplicity, low cost, and rapid execution. However, its results are often misinterpreted when extrapolated to industrial systems. This critical review examines the capabilities and limitations of bottle roll tests, with emphasis on their role in representing intrinsic chemical kinetics rather than full process behavior. Key discrepancies between laboratory and industrial conditions are analyzed, including particle-size effects, the absence of hydraulic constraints, limited representation of secondary-phase formation, and underestimation of acid consumption. Comparative analysis shows that bottle roll tests can achieve 85–95% extraction within 24–72 hours, whereas heap leaching systems typically reach 60–75% over 120–180 days, reflecting the influence of mass transfer, hydrodynamics, and time-dependent phenomena. The review highlights common scale-up pitfalls, particularly the misuse of kinetic fitting and laboratory acid consumption data for process design. A structured framework integrating bottle roll, column, and pilot testing is proposed to improve predictive capability. Data gaps are identified in coupled kinetic–transport modeling, long-term datasets, and mineralogical integration. It is concluded that bottle roll testing should be used as a screening and benchmarking tool, not as a standalone predictive method, and must be combined with scale-representative testing for reliable process evaluation.

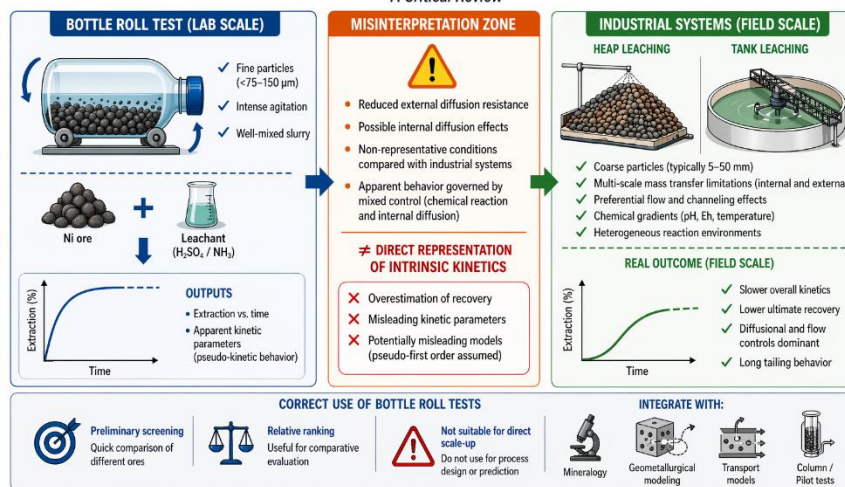
Keywords: Bottle roll testing; Heap leaching; Leaching kinetics; Scale-up; Acid consumption; Mass transfer.

Highlights

- Bottle roll tests measure intrinsic kinetics but do not represent industrial leaching systems.
- Particle size, hydraulics, and transport limitations control heap performance.
- Acid consumption and recovery are systematically misestimated at laboratory scale.
- Integrated testing (bottle roll + column + pilot) is essential for reliable scale-up.

Bottle Roll Tests for Nickel Ores: Capabilities, Limitations, and Misinterpretations

A Critical Review



1. Introduction

Leaching kinetics is a controlling factor in the technical and economic viability of hydrometallurgical operations. It directly determines residence time, reagent consumption, equipment sizing, and, consequently, both capital expenditure (CAPEX) and operating expenditure (OPEX) (Nicol, 2022; Winarko et al., 2023). In heap leaching systems, slow reaction rates impose long operational cycles, typically ranging from 100 to 200 days, which significantly increase pad footprint and delay cash flow generation (Lizama, 2021; Zhang et al., 2024). Even moderate deviations in kinetic assumptions can therefore lead to substantial design and economic errors.

Bottle roll testing is widely used as a preliminary tool for kinetic evaluation due to its low cost, simplicity, and rapid execution. Laboratory tests typically generate extraction curves within 24–72 hours using small sample masses (<math><1\ \text{kg}</math>), controlled chemical conditions, and continuous agitation (Walder et al., 2022; Notole et al., 2024). These characteristics make the method particularly attractive for early-stage screening, comparative ore evaluation, and sensitivity analysis.

Despite its widespread use, the representativeness of bottle roll testing for industrial heap leaching remains fundamentally limited. The method imposes fully mixed conditions, thereby eliminating external mass-transfer resistance and suppressing spatial gradients in pH, redox potential, and reagent concentration. In contrast, heap leaching systems operate under unsaturated flow, where fluid distribution, diffusion through porous media, and local chemical

heterogeneities govern overall performance (Moyo et al., 2025; Robertson et al., 2023). These differences are not marginal; they define the dominant rate-controlling mechanisms.

As a result, bottle roll tests frequently overestimate metal extraction rates and underestimate acid consumption when extrapolated to field conditions. Reported extractions above 85–90% within 48 hours in laboratory tests often correspond to significantly lower recoveries over extended periods in industrial heaps. This discrepancy is not only quantitative but also mechanistic, reflecting the transition from reaction-controlled systems to transport-limited regimes.

The central problem addressed in this review is therefore not the validity of bottle roll testing itself, but its interpretation. Bottle-roll tests measure intrinsic dissolution kinetics under ideal mixing conditions, whereas interactions among chemical kinetics, mass transfer, hydrodynamics, and secondary-phase formation govern heap-leaching performance.

The objective of this review is to critically evaluate the role of bottle roll testing in nickel laterite processing, with emphasis on its capabilities, limitations, and common misinterpretations. Particular attention is given to kinetic modeling, scale-up reliability, and the integration of laboratory tests with column and pilot-scale data.

This review contributes by critically organizing the mechanisms through which bottle roll data are misinterpreted in nickel laterite leaching and by proposing an integrated decision workflow that separates diagnostic use from predictive use. Unlike reviews focused only on leaching chemistry, this work links laboratory kinetic interpretation to mineralogical variability, acid consumption, transport limitations, column testing, and scale-up risk.

2. Methodology

This study was conducted as a critical review of leaching methodologies, with emphasis on kinetic interpretation, experimental limitations, and implications for process scale-up. The approach follows a structured and transparent literature selection process inspired by the PRISMA 2020 framework, but it does not aim to meet all requirements of a formal systematic review. Instead, the methodology is adapted to reflect the interpretative and mechanism-oriented nature of engineering reviews.

The objective was not to perform a statistical meta-analysis, but to critically evaluate how bottle roll data are generated, interpreted, and misapplied in the context of nickel laterite leaching.

2.1. Literature search

A structured literature search was performed across major scientific databases, including Scopus, Web of Science, ScienceDirect, SpringerLink, and Google Scholar.

The search covered publications from 2020 to 2026, with selective inclusion of earlier foundational studies where required to support mechanistic interpretation.

Search strings combined terms related to leaching kinetics, laboratory testing, and scale-up, including:

- a. *bottle roll test*
- b. *leaching kinetics*
- c. *heap leaching*
- d. *column leaching*
- e. *mass transfer*
- f. *hydrometallurgy*
- g. *nickel laterite leaching*

Boolean operators (AND, OR) were used to refine results depending on the database.

The initial search identified **233 records**.

2.2. Screening and selection

The selection process followed a structured multi-stage approach.

Duplicate removal excluded 48 records, leaving 185 studies for screening.

Stage 1 – Title and abstract screening

A total of 92 studies were excluded based on:

- a. lack of relevance to leaching processes
- b. focus on non-mineral systems
- c. absence of kinetic or transport data
- d. review papers without primary or technical contribution

This resulted in 93 studies for full-text evaluation.

Stage 2 – Full-text eligibility assessment

A further 20 studies were excluded due to:

- a. insufficient quantitative data
- b. incomplete methodological description
- c. lack of relevance to scale-up or kinetic interpretation

- d. redundancy with other selected studies

The final dataset consisted of 73 studies.

2.3. Data extraction and analysis

For each selected study, key technical variables were systematically extracted:

- a. leaching method (bottle roll, column, heap, hybrid)
- b. kinetic modeling approach (e.g., shrinking core, first-order, empirical fits)
- c. operating conditions (particle size, temperature, pH, acid concentration, time)
- d. mass transfer characteristics (diffusion regime, transport limitations)
- e. mineralogical and geochemical behavior (phase transformations, reprecipitation)

The analysis prioritizes **mechanistic interpretation over purely statistical correlation**, focusing on how experimental design influences kinetic outcomes and their extrapolation to industrial systems.

2.4. Scope of evidence and cross-system analogies

The primary focus of this review is nickel laterite leaching systems, particularly limonitic and saprolitic ores.

However, due to the limited availability of fully integrated industrial datasets, evidence from other leaching systems (e.g., copper and gold heap leaching) was selectively incorporated to support:

- a. transport phenomena
- b. hydrodynamic behavior
- c. scale-up principles

These data were used as mechanistic analogies rather than direct quantitative equivalents. Quantitative ranges derived from non-nickel systems were not treated as universally transferable and are explicitly interpreted within their context.

2.5. Methodological quality considerations

This review does not assign formal quality scores to individual studies. However, the interpretation of results explicitly considers key methodological aspects that influence data reliability:

- a. particle size and representativeness
- b. control of pH, temperature, and redox potential
- c. duration of testing

- d. presence of replicates or uncertainty analysis
- e. extent of mineralogical characterization
- f. comparison with column or pilot-scale data

Studies lacking sufficient experimental transparency or quantitative robustness were either excluded or interpreted with caution.

2.6. Limitations of the review methodology

Several limitations inherent to the present review should be acknowledged:

- a. heterogeneity of ore types, including variations in mineralogy and gangue composition
- b. variability in experimental conditions, limiting direct comparison across studies
- c. absence of formal meta-analysis, due to inconsistent reporting of quantitative data
- d. limited availability of industrial-scale datasets, particularly for nickel laterites
- e. use of cross-system analogies, which may not fully represent lateritic behavior

These limitations constrain the generalization of quantitative ranges and reinforce the need for careful interpretation of reported values.

2.7. PRISMA flow

The study selection process is summarized in Figure 1, following a PRISMA-style flow diagram.

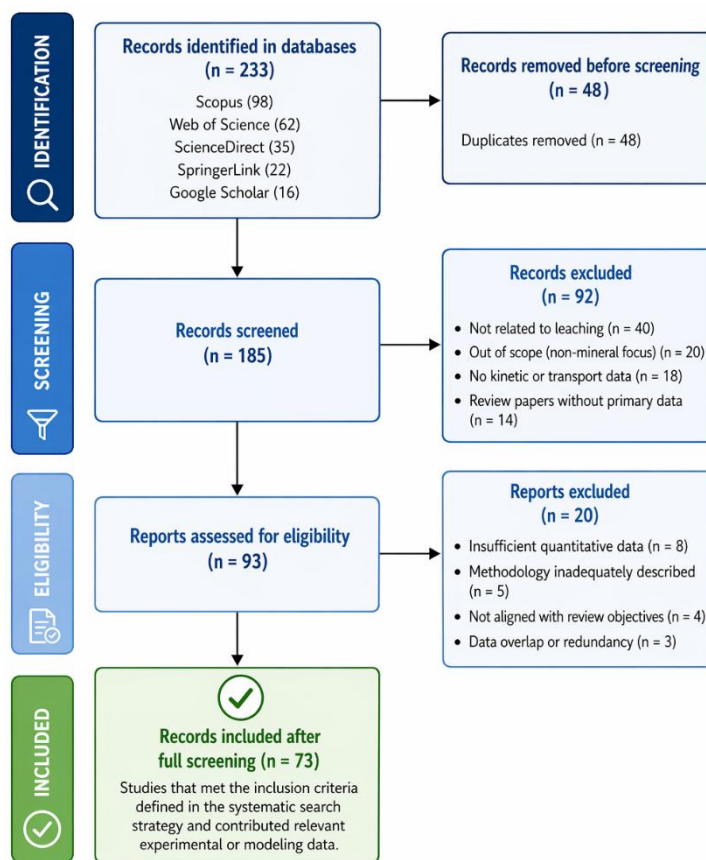


Figure 1. PRISMA-inspired flow diagram of literature identification, screening, eligibility, and inclusion. Prepared by the authors based on the PRISMA 2020 framework (Page et al., 2021).

Although this review does not perform a formal quantitative meta-analysis, the reliability of the conclusions depends strongly on the methodological quality of the underlying studies. In hydrometallurgical systems, experimental design—including particle size, control of pH and redox conditions, test duration, and mineralogical characterization—can significantly influence the interpretation of kinetics.

To improve traceability and support the hierarchy of evidence, a structured qualitative assessment of the selected studies was performed. The criteria used reflect key experimental variables known to affect leaching behavior and scale-up relevance, as summarized in Table 1.

Table 1. Methodological quality matrix of selected studies included in the review, considering key experimental parameters affecting kinetic interpretation and scale-up reliability. Prepared by the authors based on extracted data from the reviewed literature

Study group/evidence type	Ore/system focus	Particle size reported	pH, Eh, and temperature control	Test duration	Replicates/variability	Residue characterization
Bottle roll nickel laterites	Limonite and saprolite	Usually reported ; often	pH and temperature are	Short, typically	Variable; often limited	Often incomplete

		fine fraction sizes	generally reported; Eh is less frequent	24–96 h		
Column leaching studies on nickel laterites	Limonite, saprolite, blended laterites	Reported: closer to heap size	pH and temperature reported; Eh variable	Weeks to months	Usually limited	More frequent than bottle roll
Heap or pilot-scale nickel laterite studies	Lateritic ore bodies	Coarse ore, industrial PSD	Operational monitoring; spatial control limited	Months	Limited by field variability	Often partial
Kinetic modeling papers	Laterites and other mineral systems	Usually reported	Depends on dataset; often simplified	Usually short to medium	Often absent	Rarely detailed
Acid consumption studies	Laterites, silicate-rich ores	Reported in most cases	pH and acid concentration reported; Eh often absent	Short to long	Variable	Sometimes included
Secondary phase/passivation studies	Laterites and acidic leaching systems	Often reported	pH and temperature usually reported	Medium to long	Variable	Stronger when XRD/SEM/EDS included
Heap leaching transport studies from non-nickel systems	Copper, gold, and other heaps	Usually reported	Operational variables reported	Long-term or modeled	Variable	Not always relevant to Ni
General hydrometallurgical review papers	Multiple ore systems	Not applicable	Not applicable	Not applicable	Not applicable	Not a

Table 1 highlights the variability in experimental rigor across the selected studies. While some investigations provide detailed control of operating variables and include mineralogical characterization and validation against column or pilot data, others rely on simplified experimental designs with limited reporting.

This heterogeneity has direct implications for the interpretation of kinetic results. Studies based on fine particle sizes, short durations, and incomplete solid-phase analysis tend

to overrepresent intrinsic kinetics while underrepresenting transport limitations and secondary phase formation.

Therefore, conclusions drawn in this review consider not only reported outcomes but also the methodological robustness of each study. Greater weight is given to studies that incorporate mineralogical characterization, long-term testing, and comparison with scale-representative systems.

3. Fundamentals of Bottle Roll Testing

Bottle roll testing is a batch leaching method to evaluate dissolution under controlled lab conditions. It uses a closed reactor in which solid particles are immersed in a lixiviant and continuously agitated. This promotes rapid mixing, reduces mass-transfer resistance, and creates homogeneous reaction conditions (Jia et al., 2024; Ubaldini, 2021).

The method is popular in early-stage metallurgical testing because of its low cost, minimal sample requirements, and rapid results. A typical test uses less than 1 kg and takes 24–96 hours, unlike column tests, which require weeks or months and larger samples (Notole et al., 2024; Fagan-Endres et al., 2023).

3.1. Typical Configuration

Bottle roll tests follow a relatively standardized configuration, although variations exist depending on ore type and reagent system. Typical operating ranges include:

- a. Particle size: <2 mm, frequently <150 μm for kinetic studies
- b. Solid-to-liquid ratio (S/L): 1:1 to 1:3 (kg/L), occasionally up to 1:5
- c. Agitation: 30–60 rpm (rotational bottle systems)
- d. Temperature: 20–90 $^{\circ}\text{C}$, depending on system chemistry
- e. Test duration: 24–96 h, with sampling intervals of 2–12 h

(Faraji et al., 2022; Apua & Madiba, 2021; Estay & Díaz-Quezada, 2020)

Particle size is one of the most influential parameters. Fine particles (<150 μm) can increase the specific surface area by more than one order of magnitude relative to heap-leaching conditions (typically 5–50 mm), thereby directly accelerating apparent dissolution rates (Ram et al., 2020; Garcés-Granda et al., 2020).

Continuous agitation keeps particles suspended and evenly exposed to the lixiviant, eliminating stagnant zones and reducing boundary layer thickness. This makes external diffusion resistance negligible and reaction rates near intrinsic kinetics (Faraji et al., 2022).

Temperature control is also critical. For acid leaching systems, increasing temperature from 25 °C to 60 °C can increase dissolution rates by a factor of 2–5, depending on activation energy (typically 20–60 kJ/mol for lateritic systems) (He et al., 2022; Santos et al., 2021).

Periodic sampling is performed to generate kinetic curves. Liquid samples are typically filtered and analyzed for metal concentration, while solid residues are rarely characterized in detail. This imbalance between liquid- and solid-phase analyses represents a limitation in mechanistic interpretation.

To understand the implications of bottle roll testing, examine its physical setup and principles. It involves chemical conditions and mechanical agitation for solid–liquid interaction. Unlike percolation systems with stationary beds, bottle-roll tests keep particles in motion to ensure uniform lixiviant exposure.

This configuration eliminates stagnant zones and significantly reduces external mass transfer resistance. As a result, the system approaches ideal mixing conditions, where concentration gradients in the liquid phase are minimal. While this enhances reproducibility and accelerates reaction rates, it also creates a reaction environment that differs fundamentally from industrial leaching systems. The key components and operational sequence of a bottle roll test are illustrated in Figure 2.

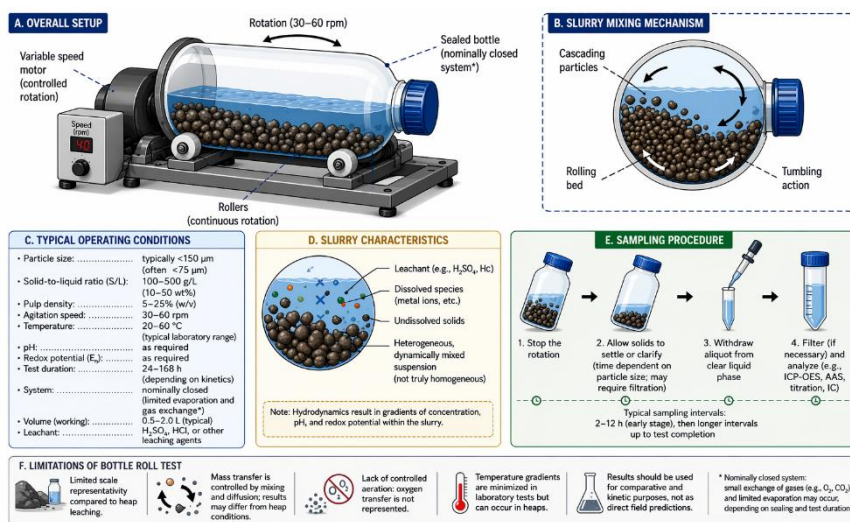


Figure 2. Schematic representation of a bottle roll test system, including rotation mechanism, slurry mixing, and sampling procedure. Adapted from (Chetty et al., 2023; Notole et al., 2024).

Figure 2 highlights the defining characteristics of bottle roll testing. The rotation mechanism generates a cascading and tumbling motion of particles, producing a well-mixed slurry in which solid particles are continuously exposed to fresh lixiviant. Under these

conditions, boundary layer thickness around particles is minimized, and external diffusion resistance becomes negligible.

The sampling procedure supports system control by using periodic liquid withdrawals to generate detailed extraction curves. However, it primarily captures dissolved species, whereas changes in the solid phase—such as passivation or pore evolution—are seldom monitored in real time.

From a mechanistic perspective, the figure lacks flow gradients and particle immersion, omitting key phenomena such as preferential flow, channeling, unsaturated conditions, and chemical gradients in pH, redox, and reagent concentrations that affect industrial leaching.

The bottle-roll test behavior exhibits a simplified kinetic regime driven primarily by chemical reaction rates. Conversely, industrial systems are under mixed control, with transport and hydrodynamics affecting performance. Recognizing this difference is essential for interpreting data and scaling processes.

3.2. Controlled Variables

Bottle roll tests allow precise control of several chemical and physical variables. The most relevant include:

- a. pH: Typically $1-2$ in acid leaching systems
- b. Acid concentration: 10–100 g/L H_2SO_4 (or equivalent for HCl systems)
- c. Temperature: Controlled within $\pm 1-2$ °C
- d. Particle size distribution: Narrow, often pre-classified
- e. Leaching time: Defined and reproducible
- f. Redox potential (Eh): Sometimes monitored but rarely controlled (Faraji et al., 2022; Ubaldini, 2021; Apua & Madiba, 2021)

Among these variables, acid concentration and pH are closely linked. In laterite leaching, acid use can surpass 200–400 kg H_2SO_4 per ton of ore due to reactions with minerals like carbonates, clays, and iron oxides (Ortega-Tong et al., 2023; Bázquez-Martell et al., 2025).

Granulometry affects kinetics and acid consumption: finer particles accelerate dissolution but increase acid demand due to more reactive gangue. This dual effect is often overlooked in simple kinetic analyses (Garces-Granda et al., 2020).

Time is treated as an independent variable in bottle roll tests, but its interpretation is often misleading. Rapid extraction within 24–48 h does not necessarily translate to similar performance in industrial systems, where transport limitations dominate.

Table 2 compares bottle-roll testing and industrial heap leaching, focusing on scale and mechanisms. Laboratory systems rely on reaction kinetics in mixing and with small particles, whereas heap leaching depends on fluid flow, diffusion, and heterogeneous reactions.

Table 2. Typical operating ranges and controlled variables in bottle roll testing compared to industrial heap leaching conditions. Adapted from (Faraji et al., 2022; Petersen & van Staden, 2025).

Parameter	Bottle Roll Testing (Lab Scale)	Heap Leaching (Industrial Scale)	Key Implication for Interpretation
Particle size	<2 mm (typically <75–150 μm)	5–50 mm (crushed ore)	Strong overestimation of kinetics due to a higher surface area
Solid-to-liquid ratio (S/L)	1:1 to 1:3 (kg/L)	~0.3–0.6 (irrigation-based, non-immersed)	Full immersion vs partial wetting leads to unrealistic contact efficiency
Agitation/flow regime	Continuous mixing (30–60 rpm)	Percolation (Darcy flow, unsaturated)	External mass transfer is minimized vs transport-limited regime
Temperature	Controlled (20–90 °C, ±1–2 °C)	Variable (ambient to ~60 °C, gradients present)	No thermal gradients in lab vs strong spatial variability in heap
pH control	Constant or tightly controlled	Variable (pH gradients along heap height)	Acid front evolution not captured in bottle roll
Acid concentration	Fixed initial (10–100 g/L H ₂ SO ₄)	Dynamic (decreases with depth and time)	Underestimation of total acid consumption
Redox potential (Eh)	Often stable or unmonitored	Spatially variable (oxygen availability, microbial activity)	Oxidation-reduction limitations ignored in lab
Test duration	24–96 h	100–200 days (typical heap cycle)	Time compression masks long-term limitations
Mass transfer regime	Reaction-controlled (minimal diffusion limitation)	Diffusion and flow controlled	Different dominant rate-controlling mechanisms
Fluid distribution	Homogeneous	Heterogeneous (channeling, dead zones)	Overestimation of leaching uniformity
Reprecipitation	Limited (closed system, short time)	Significant (jarosite, silica gel, Fe hydroxides)	Passivation effects underestimated
Permeability effects	Not applicable	Critical (compaction, fines migration)	Hydraulic limitations were not captured
Sampling	Periodic, controlled (2–12 h intervals)	Indirect (solution ponds, limited spatial resolution)	Loss of spatial resolution in industrial monitoring
Scale of operation	<1 kg sample	10 ⁵ –10 ⁷ tons ore	Scale-dependent phenomena not represented

Typical Ni extraction	80–95% (24–72 h)	60–75% (120–180 days)	Systematic overestimation of recovery
Acid consumption	Lower apparent (instant mixing)	Higher real consumption (gangue + reprecipitation)	Underestimation of OP

The table emphasizes the contrast between controlled laboratory conditions and the variability of field operations. In heap leaching, parameters such as pH, temperature, and reagent concentration vary spatially and temporally, often within wide ranges.

Among the parameters listed, particle size, flow regime, and acid distribution have the strongest impact on interpretation. Fine particles used in bottle roll tests can increase specific surface area by more than one order of magnitude, artificially accelerating dissolution rates. Similarly, continuous agitation suppresses diffusion limitations, leading to kinetic regimes that are rarely achieved in field operations.

The discrepancy in time scale is also critical. Laboratory tests compress processes occurring over months into hours, masking slow phenomena such as secondary phase formation and permeability loss. As a result, direct extrapolation of bottle roll data to industrial systems introduces systematic bias, particularly in estimating recovery and acid consumption.

Critical Assessment

The main strength of bottle roll testing lies in its ability to isolate chemical effects under controlled conditions. However, this same feature introduces a fundamental limitation. By eliminating spatial gradients and minimizing transport resistance, the method creates a reaction environment that does not represent industrial systems.

In practical terms, bottle roll tests compress multiple interacting phenomena—reaction, diffusion, and flow—into a single apparent kinetic response. This simplification is useful for screening but problematic for process design and scale-up.

4. What Bottle Roll Actually Measures

Bottle roll tests generate kinetic data that are often interpreted as representative of leaching performance. However, the method isolates only a subset of the phenomena governing metal extraction. The response measured in these tests reflects a simplified system dominated by chemical reactions under intense mixing conditions.

Under continuous agitation, solid particles are fully suspended and uniformly exposed to the lixiviant. This configuration minimizes external mass transfer resistance and promotes

rapid dissolution. As a result, bottle roll tests primarily measure intrinsic solid–liquid reaction kinetics, in which the rate is controlled by surface reactions or, in some cases, by intraparticle diffusion (Faraji et al., 2022; Ubaldini, 2021).

The method also provides a reliable estimate of initial acid consumption, particularly during the early stages of leaching. In acid systems, fast reactions with readily soluble phases—such as carbonates, amorphous oxides, and fine silicates—occur within the first hours of testing. Reported acid consumption rates in this stage can exceed 50–70% of total consumption within the first 24 h, depending on ore composition (Surimbayev et al., 2024).

In addition, bottle-roll tests determine the maximum extractable fraction under ideal chemical conditions. Because particles are fully immersed and continuously mixed, limitations related to fluid access are removed. This allows the system to approach its thermodynamic or near-equilibrium extraction limit within relatively short times. For nickel laterites, this often results in apparent recoveries above 80–90% within 48–72 h (León et al., 2025; Astuti et al., 2024).

Despite these advantages, the method does not capture the dominant mechanisms controlling industrial leaching systems. The absence of spatial gradients and flow constraints removes several key phenomena:

- a. Percolation and flow distribution, which govern liquid access to reactive surfaces
- b. Diffusion through packed beds, including both inter-particle and intra-particle transport
- c. Channeling and preferential flow, leading to uneven reagent distribution
- d. Compaction and permeability loss, affecting long-term hydraulic behavior
- e. Reprecipitation under chemical gradients, including formation of secondary phases such as jarosite or silica gel

(Liu et al., 2021; Maghsoudy et al., 2022; Robertson et al., 2023)

These omissions are significant. In heap leaching, mass transfer and hydrodynamics often control extraction rates. Effective diffusivities in packed beds can be much lower than in well-mixed systems, shifting the control from reaction to transport (Liu et al., 2025). Also, uneven irrigation can cause local reagent depletion, despite adequate overall solution chemistry.

The interpretation of bottle roll results requires a clear understanding of the fundamental differences between laboratory and industrial leaching systems. Although bottle roll tests are

often used to infer kinetic behavior, they operate under conditions that simplify or eliminate key physical processes present in heap leaching.

Bottle-roll systems ensure uniform mixing and full particle immersion, removing spatial gradients in flow, reagent concentration, and redox conditions. In contrast, heap leaching involves unsaturated flow through a porous medium, with evolving mass transfer, permeability, and heterogeneity. These differences shift the dominant mechanisms from reaction-controlled in the lab to transport-limited in the field. Figure 3 compares bottle-roll testing and heap leaching, highlighting differences in conditions, mass transfer, and control.

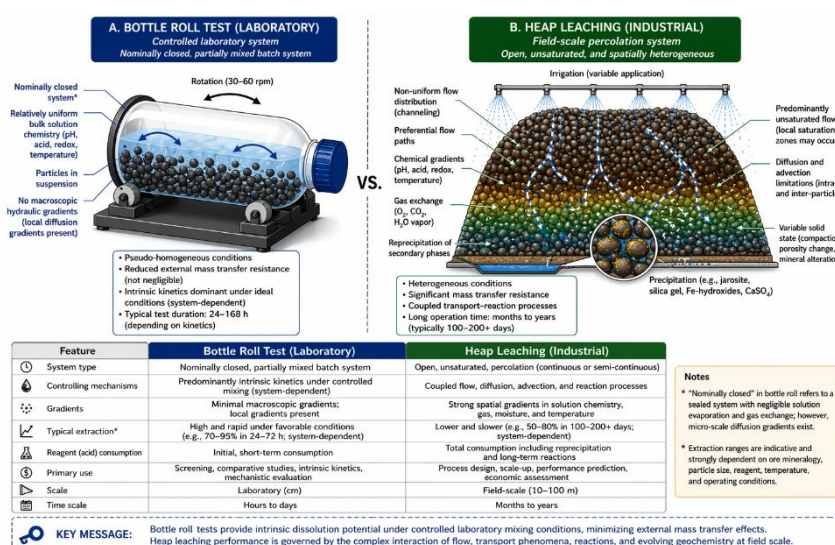


Figure 3. Conceptual comparison between bottle roll and heap leaching systems. Bottle roll represents a fully mixed system, while heap leaching is governed by coupled flow, diffusion, and reaction processes. Adapted from (Fernando et al., 2020; Robertson et al., 2023).

Figure 3 illustrates the fundamental difference between the two systems. In bottle-roll tests, the slurry behaves as a homogeneous medium with a uniform chemical potential. In contrast, heap leaching systems exhibit strong spatial variability, where fluid flow, gas transport, and solid structure interact dynamically.

This distinction impacts data interpretation. Bottle-roll results show apparent kinetics under ideal conditions, not the effective kinetics in industrial systems. Moving from lab to field adds resistance, reducing extraction speed and increasing reagent use.

A key consequence is the overestimation of performance when bottle roll data are used without correction. High lab extraction rates may not be attainable in practice due to transport limitations and changes in mineralogy. Thus, bottle roll testing indicates chemical potential, not operational performance.

5. Reaction Mechanisms in Nickel Ores

Nickel laterite ores show complex behavior during leaching, influenced by nickel distribution in iron oxides and silicates and operating conditions. Multiple dissolution pathways occur simultaneously with different rate controls.

5.1. Lateritic Systems (H₂SO₄ and HCl Leaching)

Laterites are divided into limonitic and saprolitic zones. In limonitic ores, nickel mainly substitutes within goethite (FeOOH), while in saprolitic ores, it associates with Mg-rich silicates like serpentine and olivine (Astuti et al., 2024; He et al., 2022).

Nickel laterite ores are typically classified into limonitic and saprolitic domains, which exhibit fundamentally different mineralogical compositions and leaching behavior. These differences directly influence kinetic response, acid consumption, secondary phase formation, and scale-up reliability.

To clarify these distinctions and their implications for interpreting bottle roll test results, a comparative analysis is presented in Table 3.

Table 3. Comparative behavior of limonitic and saprolitic nickel laterite ores in bottle roll and heap leaching systems, highlighting implications for kinetic interpretation, acid consumption, and scale-up reliability. Prepared by the authors based on literature synthesis.

Parameter	Limonitic laterites	Saprolitic laterites	Implication for bottle roll interpretation
Main Ni-hosting phase	Ni incorporated in goethite (FeOOH)	Ni associated with Mg-silicates (serpentine, olivine)	Different dissolution mechanisms not captured equally in short tests
Dissolution mechanism	Relatively fast acid dissolution of Fe oxyhydroxides	Slower dissolution of Mg-silicates, often diffusion-limited	Bottle roll overrepresents limonite kinetics
Typical kinetics (lab)	Fast extraction (often >80% in 24–48 h)	Moderate extraction, slower response	Saprolite kinetics more sensitive to time and diffusion
Typical kinetics (heap)	Moderate recovery with potential reprecipitation effects	Slower recovery, strongly transport-limited	Scale-up error larger for saprolitic systems
Acid consumption	Moderate to high (Fe dissolution, hydrolysis)	Very high (Mg-silicate dissolution)	Bottle roll underestimates acid demand in saprolites
Secondary phase formation	Fe hydroxides, jarosite	Silica gel, Mg-rich phases	Bottle roll underrepresents long-term passivation
Passivation risk	Moderate (Fe reprecipitation)	High (silica gel formation, pore blocking)	Strong underestimation in laboratory tests

Permeability impact	Moderate reduction over time	Severe reduction due to silica gel formation	Not captured in bottle roll tests
Sensitivity to particle size	Moderate	Very high	Fine grinding artificially enhances saprolite performance
Sensitivity to acid distribution	Moderate	High	Bottle roll masks spatial acid depletion in saprolites
Transport limitation relevance	Moderate	Critical	Saprolites require column/pilot validation
Reliability of bottle roll for prediction	Limited	Very low	Direct extrapolation highly unreliable for saprolites
Recommended testing pathway	Bottle roll → column → pilot	Bottle roll → long-term column → pilot	Saprolites require extended validation

Table 3 shows that limonitic and saprolitic laterites respond fundamentally differently to leaching conditions. Limonitic ores, dominated by goethite, typically exhibit faster initial dissolution under acidic conditions, which is relatively well captured in bottle roll tests. However, reprecipitation of iron phases can reduce recovery over time, particularly in industrial systems.

In contrast, saprolitic ores are controlled by the dissolution of Mg-silicates, which is slower, more acid-intensive, and often diffusion-limited. These systems are highly sensitive to particle size, permeability, and acid distribution, making bottle roll tests particularly unreliable for predicting industrial performance.

The table also highlights that secondary phase formation—especially silica gel in saprolitic systems—can significantly reduce permeability and alter long-term kinetics—effects that are not captured by short-duration laboratory tests.

From a scale-up perspective, these differences suggest that bottle-roll tests may provide reasonable screening information for limonitic ores but are insufficient for predictive purposes in saprolitic systems without extended column- or pilot-scale validation.

In acidic leaching systems, the main reactions include:

- a. Dissolution of goethite, releasing Ni^{2+} and Fe^{3+} into solution
- b. Dissolution of Mg-silicates is typically slower and often diffusion-limited
- c. Formation of amorphous silica, which may precipitate and affect permeability (Li et al., 2020; Top et al., 2020; Shayakhmetova et al., 2025)

The goethite's dissolution is quick under strong acids (pH <1–2), especially at 40–90 °C. Activation energies for iron oxide vary from 25 to 60 kJ/mol, reflecting mixed kinetic control based on mineral structure and porosity (He et al., 2022; Santos et al., 2021).

Silicate dissolution is slower, requiring longer contact times and more acid—often 300–500 kg H₂SO₄ per ton of ore in lateritic systems—raising costs, especially when acid distribution is uneven (Thomas, 2021).

Lateritic leaching forms secondary phases such as amorphous silica and iron hydroxides, which coat particles or block pores, thereby reducing diffusivity and slowing metal extraction (Stanković et al., 2022).

Figure 4 summarizes how mineralogy influences nickel extraction from lateritic ores during acidic leaching. It differentiates limonitic and saprolitic systems, showing nickel's distribution in Fe oxyhydroxides and Mg-silicates. The diagram highlights key reaction mechanisms—goethite dissolution, silicate breakdown, and secondary phases—focusing on their kinetic behaviors and effects on acid use and mass transfer. Linking mineralogy to reaction pathways, it offers a mechanistic view of leaching performance beyond simple kinetic models.

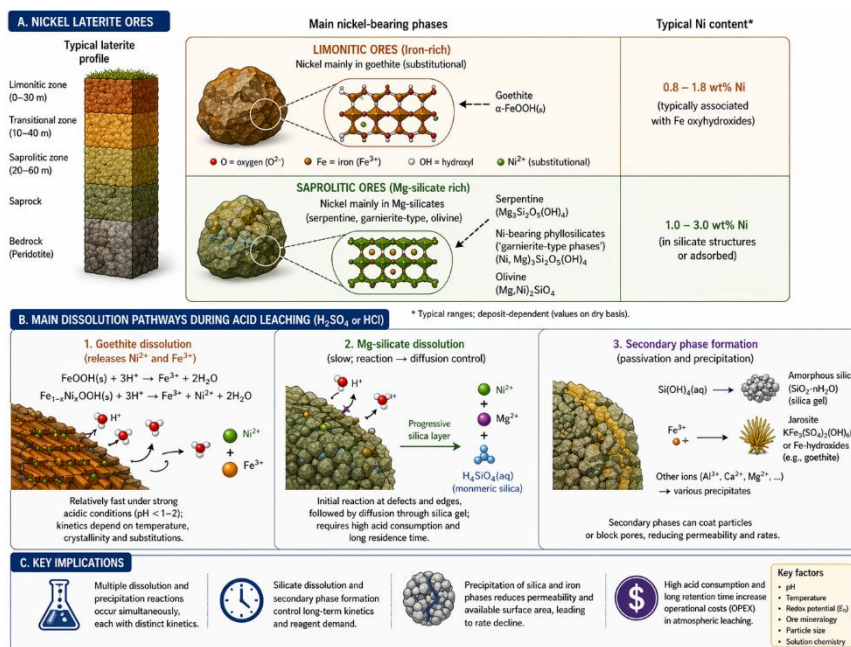


Figure 4. Schematic representation of nickel distribution in lateritic ores and main dissolution pathways during acid leaching. Includes goethite dissolution, silicate breakdown, and secondary phase formation. Adapted from (Astuti et al., 2024; Li et al., 2020).

The figure illustrates that nickel extraction is governed not by a single reaction but by interactions among multiple mineral phases and evolving solid–liquid interfaces.

5.2. Kinetic Control Regimes

Leaching kinetics in lateritic systems can follow different rate-controlling mechanisms, depending on mineralogy, particle size, and hydrodynamic conditions. The main regimes include:

- a. Surface reaction control, where the chemical reaction at the solid–liquid interface is rate-limiting
- b. Intra-particle diffusion control, where diffusion through pores or product layers limits transport
- c. Film diffusion control, where mass transfer across the liquid boundary layer controls the rate

(Wanta et al., 2020; Kang et al., 2026; Nasab et al., 2020)

In well-mixed systems such as bottle roll tests, external film resistance is minimized. The boundary layer thickness is reduced, and fresh reagent is continuously supplied to the particle surface. Under these conditions, the system tends to operate under apparent reaction-controlled kinetics, even when diffusion limitations would be significant under field conditions.

In contrast, heap leaching systems typically operate under mixed or diffusion-controlled regimes. Effective diffusivity in packed beds is reduced by tortuosity and partial saturation, often by one to two orders of magnitude compared to fully mixed systems (Liu et al., 2025; Maghsoudy et al., 2022). This regime shift directly affects extraction rates and time scales.

Quantitatively, this transition can be illustrated by comparing characteristic times:

- a. Bottle roll tests: characteristic reaction time <24–72 h
- b. Heap leaching: characteristic transport-controlled time 100–200 days

(León et al., 2025; Yilmaz et al., 2020)

Critical Assessment

A central limitation of bottle roll testing is its tendency to force a kinetic regime that does not occur in industrial systems. By eliminating external mass transfer resistance and enhancing particle exposure, the method biases the system toward reaction-controlled behavior. This leads to an overestimation of intrinsic kinetics and masks the role of diffusion and flow limitations.

In practical terms, the kinetic parameters derived from bottle roll tests are not directly transferable to heap leaching design. Apparent rate constants obtained under ideal mixing

conditions do not account for transport constraints, phase transformations, or evolving permeability.

Therefore, the interpretation of kinetic data must consider the transition from laboratory to field conditions. Reaction mechanisms identified in bottle roll tests remain valid at the microscale, but their macroscopic expression is strongly modified by transport phenomena and system heterogeneity.

6. Mathematical Modeling of Bottle Roll Data

Mathematical modeling is commonly applied to bottle roll data to extract kinetic parameters and to support process interpretation. However, the validity of these models depends strongly on the assumptions embedded in their formulation. Because bottle roll tests operate under ideal mixing conditions, the resulting kinetic parameters often reflect simplified systems that do not represent industrial leaching environments.

The most frequently used models include:

- a. Shrinking core model (SCM)
- b. First-order kinetic models
- c. Empirical logarithmic fits (log t relationships)

(Kurniawan et al., 2024; Raschman et al., 2025)

The shrinking core model is widely used because of its physical interpretation. It assumes that leaching proceeds from the particle surface inward, with a shrinking unreacted core. Depending on the controlling step, the model can describe reaction-controlled, diffusion-controlled, or mixed regimes. In bottle-roll tests, the model often fits the data assuming surface-reaction control, with linear relationships between conversion and time.

First-order kinetic models are also commonly used, particularly for rapid screening. These models assume that the dissolution rate is proportional to the remaining unreacted fraction. Although simple, they lack mechanistic depth and are sensitive to experimental noise.

Empirical logarithmic fits are frequently used when data deviates from classical models. These fits can produce high correlation coefficients ($R^2 > 0.95$), but they provide limited physical insight into the underlying mechanisms.

Kinetic modeling is widely used to interpret bottle roll test data and to infer rate-controlling mechanisms in leaching systems. In practice, experimental extraction curves are often fitted using simplified models such as the shrinking core model (SCM) or first-order

kinetics. However, different models can provide similarly high statistical fits to the same dataset, even when they are based on fundamentally different physical assumptions, as illustrated in Figure 5.

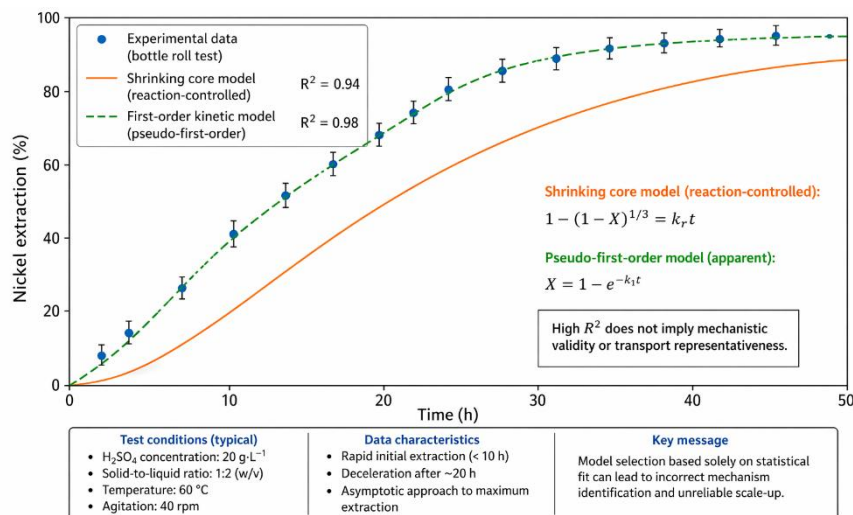


Figure 5. Typical fitting of bottle roll data using shrinking core and first-order models. High correlation does not guarantee physical validity or predictive capability. Adapted from (Wanta et al., 2020; Faraji et al., 2022).

Figure 5 illustrates a common issue in kinetic modeling: multiple models can fit the same dataset with similar statistical quality. For example, both shrinking core and first-order models may yield R^2 values above 0.95 for the same extraction curve. However, the corresponding interpretations of rate control can differ significantly.

This leads to a critical limitation. Model selection is often based on goodness-of-fit rather than on consistency with physical processes. As a result, parameters such as apparent rate constants or activation energies may not reflect actual controlling mechanisms.

Overfitting is a recurrent problem. When models are calibrated using short-duration tests (24–72 h), they capture early-stage behavior but fail to represent long-term dynamics. In lateritic systems, slow processes such as silicate dissolution, pore diffusion, and secondary phase formation become dominant over extended time scales. These effects are not captured by models fitted to early data.

A second limitation is the use of these models for direct industrial extrapolation. Parameters derived under fully mixed conditions implicitly assume negligible external mass transfer resistance. In heap leaching systems, this assumption is invalid. Effective diffusivity in packed beds is significantly reduced due to tortuosity and partial saturation, often by factors of 10–100 compared to liquid-phase diffusion (Liu et al., 2025; Maghsoudy et al., 2022). This

shift alters the dominant rate-controlling mechanism from reaction-controlled to transport-limited.

Quantitatively, this mismatch can be illustrated by comparing characteristic rate constants. Apparent rate constants derived from bottle roll tests may overestimate field-scale rates by one to two orders of magnitude. Consequently, predicted leaching times based on these models can be severely underestimated.

A further issue is the neglect of evolving system properties. Most models assume constant particle size, porosity, and surface area. In reality, these properties change during leaching due to dissolution, precipitation, and structural collapse. For example, silica precipitation can reduce pore connectivity, decreasing effective diffusivity and slowing reaction rates over time (Stanković et al., 2022).

Critical Assessment

Mathematical models applied to bottle roll data provide useful descriptors of laboratory behavior but should not be interpreted as predictive tools for industrial systems. High statistical agreement does not imply mechanistic validity. The use of simplified models for scale-up introduces systematic bias, particularly when transport limitations dominate.

For robust process design, kinetic modeling must be integrated with transport models and validated using column or pilot-scale data. Without this integration, model-based predictions risk underestimating residence time, overestimating recovery, and misrepresenting reagent consumption.

7. Granulometry and Liberation Effects

Particle size is one of the most influential variables in leaching kinetics. Grinding increases specific surface area and promotes faster dissolution rates, often leading to an overestimation of process performance under laboratory conditions (Ram et al., 2020). In bottle-roll tests, samples are typically ground to particle sizes below 150 μm , whereas industrial heap leaching uses much coarser particles, usually 5–50 mm.

This discrepancy fundamentally alters the leaching response. Fine grinding promotes artificial liberation of nickel-bearing phases that would remain partially encapsulated under heap conditions. As a result, the exposed reactive surface increases significantly, accelerating apparent kinetics and masking transport limitations such as intraparticle diffusion and solution percolation (Garces-Granda et al., 2020).

From a quantitative perspective, bottle roll tests frequently report nickel extraction levels between 85–95% within 24–72 hours. In contrast, heap leaching systems typically achieve only 60–75% extraction over 120–180 days, depending on ore type, permeability, and irrigation conditions (León et al., 2025). This difference represents not only a kinetic acceleration but a shift in the controlling mechanisms, from surface-reaction-dominated regimes in fine particles to diffusion and flow-limited regimes in coarse heaps.

The effect of particle size on leaching performance can be visualized by comparing extraction kinetics under fine- and coarse-grained conditions, as shown in Figure 6.

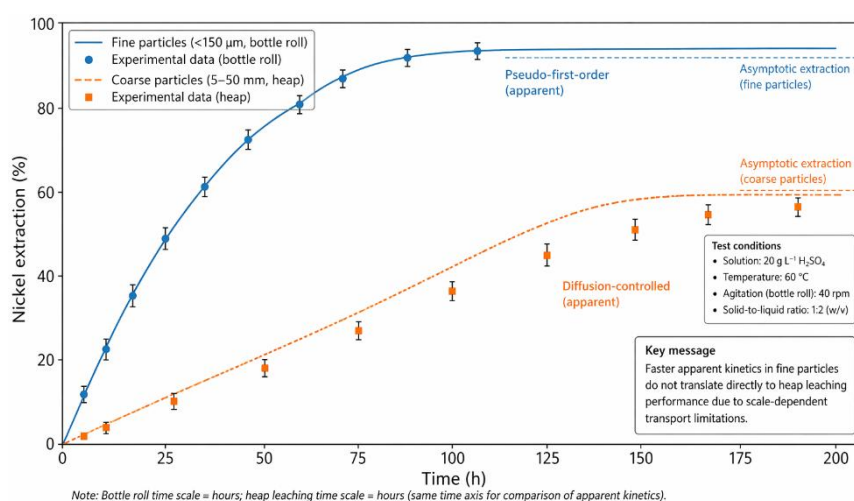


Figure 6. Effect of particle size on leaching kinetics: comparison between fine particles (<150 μm, bottle roll) and coarse particles (5–50 mm, heap leaching). Fine grinding increases apparent extraction rates due to higher surface area and artificial phase liberation. Adapted from Ram et al. (2020), Garcés-Granda et al. (2020), and León et al. (2025).

The figure shows similar extraction levels can occur across different time scales and transport regimes. Quick reactions in finely ground systems don't apply to heap leaching, where diffusion resistance, reagent access, and mineral heterogeneity drive the process.

The central limitation of bottle roll testing is its inability to preserve the ore's structural integrity. Grinding removes key physical constraints present in industrial systems, including pore connectivity, preferential flow paths, and partial mineral encapsulation. Consequently, bottle-roll results should not be interpreted as predictive of heap-leaching performance without correction factors or scale-up validation.

A typical misinterpretation occurs when bottle-roll tests indicate up to 90% extraction within 48 hours, which suggests high process efficiency. In practice, the same ore may require 120–180 days in a heap-leach operation to achieve significantly lower recovery levels. This

discrepancy has direct implications for project evaluation, particularly in estimating residence time, pad size, reagent consumption, and overall CAPEX/OPEX.

Engineering implications

From a design perspective, the use of fine-particle kinetic data without adjustment can lead to:

- a. Underestimation of heap residence time (by factors of 10–50×)
- b. Oversizing of expected recovery in feasibility studies
- c. Miscalculation of irrigation rates and solution management
- d. Underprediction of acid consumption due to increased exposure of gangue minerals

To mitigate these issues, it is essential to integrate bottle roll results with column leach tests, permeability measurements, and geometallurgical characterization. Scaling approaches should explicitly account for particle size distribution, mineral liberation, and transport limitations.

These granulometric effects are closely linked to another major source of misinterpretation: acid consumption. The increased surface area generated by grinding not only accelerates dissolution kinetics but also enhances the reactivity of gangue minerals, often leading to artificially elevated or misleading acid consumption trends.

8. Acid Consumption Misinterpretation

Acid consumption is a critical parameter in leaching design, directly affecting operating cost, reagent logistics, and environmental control. In laboratory-scale bottle roll tests, acid consumption is often underestimated due to the idealized reaction environment. Continuous agitation promotes homogeneous mixing, ensuring rapid and uniform contact between acid and reactive mineral phases (Wang et al., 2022).

Under these conditions, acid-consuming reactions occur quickly and appear to reach completion within short time frames. However, this behavior does not capture the complexity of industrial heap leaching systems, in which fluid flow, mineral heterogeneity, and time-dependent reactions play dominant roles.

In heap leaching, acid consumption is governed by spatial and temporal gradients. As the leaching solution percolates through the ore bed, the acid concentration decreases with depth, creating vertical gradients that strongly influence reaction pathways. In addition, secondary reactions—such as dissolution of gangue minerals, precipitation of intermediate

phases, and reprecipitation of dissolved species—contribute to delayed and cumulative acid consumption (Bárzaga-Martell et al., 2025).

Typical industrial observations indicate that total acid consumption in heaps can be 1.5 to 3 times higher than values estimated from bottle roll tests, depending on mineralogy, permeability, and irrigation strategy. This discrepancy is particularly relevant in lateritic and silicate-rich ores, where slow-reacting phases continue to consume acid over extended periods.

Several commonly reported quantitative ranges—like extraction efficiency, acid use, and recovery differences—are key in process evaluation. However, these are often given as general estimates without clear context, mineralogy, or scale.

To improve transparency and reduce the risk of overgeneralization, the main quantitative ranges discussed in this review are explicitly contextualized according to their source, mineral system, and operating conditions, as summarized in Table 4.

Table 4. Traceability of key quantitative ranges reported in the literature, including extraction, acid consumption, kinetic parameters, and scale-up discrepancies. Values are contextualized according to mineral system, source type, and operating conditions. Prepared by the authors based on literature synthesis.

Parameter	Reported Range	Mineral System	Source Type	Operating Conditions
Ni extraction (bottle roll)	85–95% (24–72 h)	Nickel laterites (limonite/saprolite)	Laboratory studies	Fine particles (<150 µm), full mixing
Ni extraction (heap leaching)	60–75% (120–180 days)	Nickel laterites	Industrial / pilot data	Coarse particles (5–50 mm), percolation flow
Acid consumption (laboratory)	Baseline (apparent)	Nickel laterites	Laboratory studies	Instant mixing, short duration
Acid consumption (industrial)	1.5–3× higher than lab	Nickel laterites (especially saprolitic ores)	Industrial observations / engineering estimates	Long-term leaching, spatial gradients
Residence time	24–72 h (lab) vs 120–180 days (heap)	Nickel laterites	Combined literature synthesis	Fully mixed vs transport-limited systems
Apparent rate constants	1–2 orders of magnitude higher in lab	General leaching systems	Modeling studies	Short-duration tests, fine particles

OPEX increase (acid-related)	+20–60%	Nickel laterites	Engineering estimates	Based on acid consumption deviations
Permeability loss	20–60% reduction over time	Heap leaching systems	Industrial / pilot data	Long-term operation, secondary phases
Recovery loss due to passivation	5–20 percentage points	Lateritic systems	Literature synthesis	Secondary phase formation (silica, Fe hydroxides)
Effective diffusivity reduction	10–100× lower than liquid systems	Packed beds / heaps	Transport studies	Unsaturated flow, tortuosity

Table 4 shows that commonly cited values are strongly dependent on experimental conditions and mineralogical characteristics. Laboratory-scale results, particularly those obtained under bottle roll conditions, consistently reflect idealized systems with enhanced kinetics and limited transport constraints.

In contrast, industrial heap leaching performance is governed by coupled phenomena, including diffusion, flow distribution, and secondary phase formation. As a result, direct extrapolation of laboratory-derived values leads to systematic bias in estimating recovery, acid consumption, and residence time.

The table also highlights that some ranges are derived from engineering interpretation rather than direct measurement. These values should therefore be treated as indicative trends rather than predictive parameters.

By explicitly distinguishing between laboratory data, industrial observations, and interpretative synthesis, this review aims to improve the reliability of scale-up analysis and reduce misinterpretation of kinetic results.

The fundamental differences in acid consumption mechanisms between bottle-roll and heap-leaching systems are summarized in Table 5.

Table 5. Comparison of acid consumption mechanisms in bottle roll and heap leaching systems. Adapted from Winardhi et al. (2022) and Mokmeli (2020).

Parameter	Bottle Roll Test	Heap Leaching System	Technical Implication
Mixing regime	Fully homogeneous	Heterogeneous flow	Non-uniform reaction rates
Acid distribution	Uniform	Vertical gradients	Progressive depletion with depth
Reaction time	Short (hours–days)	Long (weeks–months)	Delayed consumption effects
Secondary reactions	Limited	Significant	Additional acid demand
Reprecipitation	Negligible	Frequent	Acid consumed without metal recovery
Channeling	Absent	Common	Uneven reagent utilization
Typical acid consumption	Baseline	1.5–3× higher	Underestimation in lab tests

Table 5 highlights that bottle roll tests fail to capture key mechanisms responsible for delayed and additional acid consumption in heap systems. In particular, the absence of concentration gradients and secondary reactions systematically leads to lower acid demand estimates in laboratory tests.

The discrepancies between bottle-roll and heap-leaching results are not only technical but also have direct economic implications. Differences in acid consumption, residence time, and recovery translate into significant deviations in both operating and capital expenditures.

Table 6 summarizes the typical differences between bottle roll testing and heap leaching conditions, highlighting their impact on process design, cost estimation, and economic performance.

Table 6. Economic implications of bottle roll vs. heap leaching data interpretation. Adapted from Close (2021), Pereira (2019), and Fernando et al. (2020), with additional interpretation and synthesis by the authors

Parameter	Bottle Roll Testing	Heap Leaching (Industrial)	Economic Impact
Acid consumption	Baseline (well-mixed conditions)	1.5–3× higher due to side reactions and reprecipitation	OPEX increase (typically +20–60%)
Residence time	24–72 h	120–180 days (or longer)	CAPEX increase (heap size, infrastructure)
Metal extraction	85–95% (apparent)	60–75% (typical industrial)	Revenue overestimation
Flow regime	Homogeneous mixing	Heterogeneous percolation	Design uncertainty and inefficiency
Secondary reactions	Limited	Significant (precipitation, passivation)	Increased reagent consumption

Mass transfer	Negligible limitations	Diffusion and transport-limited	Reduced kinetics and productivity
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As shown in Table 6, bottle roll tests systematically underestimate the complexity of industrial leaching systems. The combined effects of transport limitations, hydrodynamic heterogeneity, and secondary reactions result in lower recoveries, higher reagent consumption, and longer processing times at scale.

These differences directly impact project economics, affecting both OPEX and CAPEX, and highlight the need to integrate laboratory, column, and pilot-scale data for reliable process design.

From an economic perspective, acid consumption is a major contributor to operating expenditure (OPEX) in heap leaching systems. Typical acid costs range from 10 to 50 USD per ton of ore, depending on logistics, acid concentration, and site location.

When bottle roll data are used without correction, the systematic underestimation of acid consumption (typically by a factor of 1.5 to 3×) can lead to significant economic deviations. In practical terms, this may correspond to an additional 20–60% increase in total OPEX, particularly in lateritic ores where gangue reactions and reprecipitation are substantial.

These discrepancies are not marginal. At an industrial scale, even a deviation of 20–30 kg H₂SO₄ per ton of ore can translate into millions of dollars per year in additional reagent costs. Therefore, acid consumption derived from bottle roll tests should be treated as a lower-bound estimate and validated using column- or pilot-scale data.

Bottle roll testing cannot replicate how acid use varies over time and space. In industrial heaps, acid is consumed not only by primary reactions but also by interactions with gangue minerals and reprecipitation, which do not recover metal. These effects accumulate and are influenced by flow patterns and mineral distribution.

A common misinterpretation occurs when acid consumption measured in bottle roll tests is directly used for process design. This approach can significantly underestimate reagent requirements, leading to errors in OPEX estimation and acid supply infrastructure. In large-scale operations, even a 20–30 kg/t deviation in acid consumption can result in substantial cost impacts.

Engineering implications

- a. Failure to account for realistic acid consumption can lead to:
- b. Undersizing of acid storage and delivery systems

- c. Underestimation of operating costs (OPEX)
- d. Inefficient irrigation strategies
- e. Increased environmental risk due to uncontrolled solution chemistry

To improve reliability, acid consumption data from bottle roll tests should be complemented by column leaching experiments, long-term kinetic monitoring, and geochemical modeling that includes secondary-phase formation and reprecipitation reactions.

These effects are closely linked to the formation of secondary phases and reprecipitation phenomena, which further complicate the interpretation of leaching performance and reagent consumption.

9. Reprecipitation and Secondary Phases

The formation of secondary phases is a key factor controlling long-term leaching performance. During acid leaching, dissolved species can reprecipitate as new mineral phases, altering both reaction pathways and transport properties. Common secondary phases include jarosite, iron hydroxides, and silica gel, all of which are frequently reported in acidic leaching systems (O'Connor & Eksteen, 2020).

These phases directly affect process performance by consuming acid, removing iron, and co-precipitating valuable metals. Iron hydroxides coat mineral surfaces, limiting reagent access. Silica gel causes pore blockage and permeability reduction. These effects lead to passivation, lower metal recovery, and poor flow.

The progressive development of passivation layers and the loss of permeability can reflect the impact of secondary phase formation on leaching performance, as illustrated in Figure 7, highlighting the risk of permeability collapse and recovery loss under heap conditions.

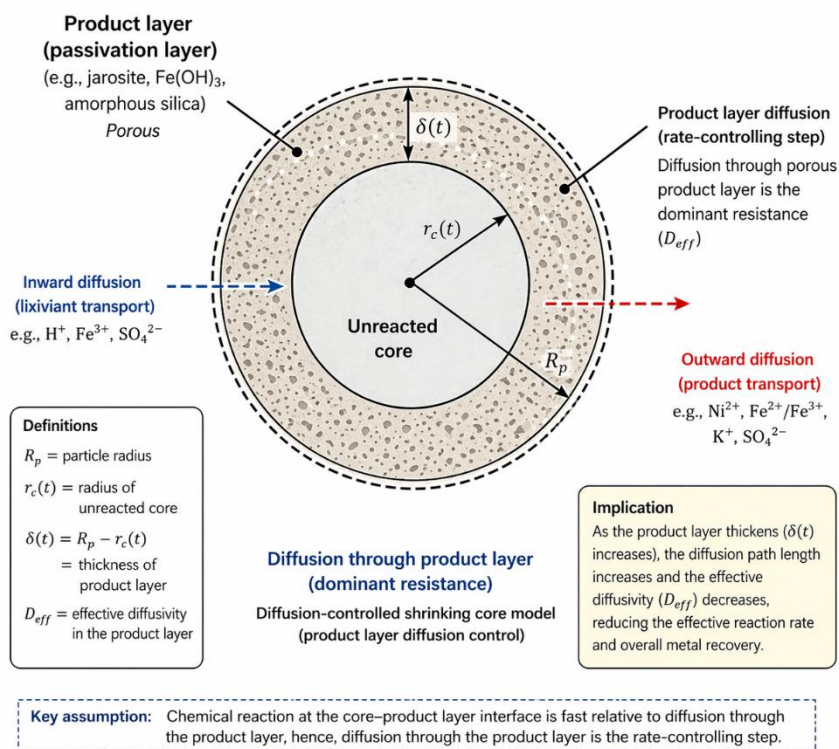


Figure 7. Formation of secondary phases during leaching: schematic representation of jarosite precipitation, iron hydroxide coatings, and silica gel formation, illustrating their impact on permeability and metal recovery. Adapted from O'Connor and Eksteen (2020) and Bare et al. (2021).

Figure 7 illustrates how secondary phases progressively alter the leaching environment. Precipitation can occur within pores, on particle surfaces, or along flow channels, thereby reducing permeability and restricting reagent transport. These effects are cumulative and become more pronounced over time.

Critical comparison: bottle roll vs. heap systems

Bottle roll tests systematically underestimate the impact of secondary phase formation. Under laboratory conditions, the system is closed, well-mixed, and characterized by short residence times. Under these conditions, dissolved species remain in solution or do not reach supersaturation levels required for significant precipitation (Bare et al., 2021).

In contrast, heap leaching systems operate under open and dynamic conditions. As the leaching solution percolates through the ore, local changes in pH, redox potential, and ion concentration promote the formation of secondary phases. Typical examples include:

- Jarosite precipitation under acidic and oxidizing conditions, consuming sulfuric acid and incorporating Fe^{3+}
- Iron hydroxide formation due to hydrolysis reactions, leading to surface passivation

- c. Silica gel formation from silicate dissolution causes pore blockage and reduced permeability

These processes are strongly time-dependent and can significantly reduce recovery over extended periods of operation.

Quantitative implications

Industrial observations indicate that permeability losses due to secondary phase formation can reduce effective solution flow rates by 20–60% over the life of a heap. In parallel, passivation effects may reduce ultimate metal recovery by 5–20 percentage points relative to initial projections from laboratory tests.

In bottle roll tests, these effects are largely absent or minimized, leading to optimistic recovery predictions and underestimation of long-term performance degradation.

Engineering implications

Failure to account for secondary phase formation can lead to:

- a. Overestimation of metal recovery
- b. Underestimation of leaching time
- c. Poor design of irrigation and drainage systems
- d. Increased acid consumption due to precipitation reactions
- e. Operational instability due to permeability loss

To address these challenges, incorporate long-term column testing, geochemical modeling, and mineralogical monitoring into the evaluation workflow. Discrepancies between lab and industrial behavior are especially critical during scale-up, where simplified assumptions from bottle roll tests are directly used as design parameters..

10. Scale-Up Pitfalls

Scale-up from laboratory bottle roll tests to industrial leaching systems remains one of the most critical sources of error in process design. Bottle roll results are often interpreted as directly representative of industrial performance, despite fundamental differences in particle size, hydrodynamics, mass transfer, and reaction environment. These misinterpretations can lead to unrealistic expectations of recovery, reagent consumption, and operational stability.

A common issue is assuming rapid lab extraction indicates process viability. In practice, shifting from fine particles under well-mixed conditions to coarse particles in percolation

systems causes strong kinetic limitations. As discussed, extraction times can rise from hours to months when moving from bottle-roll to heap-leaching conditions.

Similarly, acid consumption measured under laboratory conditions is frequently used without correction in process design. This approach neglects delayed consumption due to secondary reactions, reprecipitation, and flow heterogeneity, resulting in a systematic underestimation of operating costs (OPEX). In large-scale operations, deviations of 20–50 kg acid/t ore are not uncommon and can significantly affect economic feasibility.

The economic impact of these scale-up errors extends beyond reagent consumption. Underestimation of leaching time directly affects heap size, irrigation infrastructure, and solution management systems, leading to increased capital expenditure (CAPEX).

For example, extending residence time from laboratory-predicted values (2–3 days) to industrial conditions (120–180 days) can increase heap footprint by one order of magnitude. This directly impacts pad construction costs, stacking systems, and solution handling capacity.

Consequently, misinterpretation of bottle roll data may result not only in OPEX underestimation but also in significant CAPEX overruns and reduced project net present value (NPV).

Another recurring misinterpretation involves kinetic model fitting. High correlation between experimental data and simplified models is often taken as evidence of predictive capability. However, such fits do not guarantee that the governing mechanisms are correctly represented, particularly under conditions where mass transfer and hydraulic behavior dominate.

The most frequent scale-up misinterpretations and their industrial consequences are summarized in Table 7.

Table 7. Common scale-up errors and industrial consequences. Adapted from Van Staden and Petersen (2021)

Misinterpretation	Industrial Consequence	Technical Impact
Fast extraction = process viability	Underestimation of residence time	The heap cycle extended from days to months
Measured acid consumption = real consumption	Underestimation of OPEX	Increased reagent demand (1.5–3×)
Model fit = predictive capability	Failure during ramp-up	Incorrect control of kinetics and recovery
Homogeneous conditions assumed	Ignored flow heterogeneity	Channeling and uneven leaching
Laboratory recovery = plant recovery	Overestimation of metal output	Reduced project NPV and delayed payback

Table 7 highlights that scale-up errors are not isolated but systematic, arising from the direct transfer of laboratory assumptions to industrial systems. These errors accumulate across multiple parameters, amplifying their impact on process performance and economic outcomes.

The main challenge in scale-up is ignoring mass-transfer and hydraulic limits. In heap leaching, metal recovery depends on solution distribution, permeability, and diffusion, not reaction kinetics. Bottle roll tests, which use agitation and fine particles, bypass these limitations. Failures during commissioning and ramp-up often result from these missed constraints, leading to redesigns, increased reagents, or longer leaching times, negatively impacting economics.

Engineering implications

Failure to properly account for scale-up effects can lead to:

- a. Undersized leach pads and insufficient residence time
- b. Underestimated reagent storage and supply systems
- c. Inaccurate economic projections (NPV, IRR, payback)
- d. Operational instability during ramp-up

To reduce risk, scale-up strategies should integrate:

- Column leaching tests under realistic flow conditions
- Hydraulic characterization (permeability, percolation rates)
- Geometallurgical modeling of ore variability
- Coupled kinetic–transport models

Despite these limitations, bottle roll tests remain valuable when used within their appropriate scope. Understanding when and how to apply these tests is essential for reliable process evaluation.

11. When Bottle Roll is Useful

Despite its limitations for scale-up and process prediction, bottle roll testing remains a valuable tool when applied within a well-defined scope. Its strength lies in rapid, controlled experimentation under reproducible conditions, enabling early-stage decision-making and comparative evaluation of ores and process variables.

Bottle roll tests quickly screen leaching behavior, evaluating multiple samples under standardized conditions within 24–72 hours. They help classify ore reactivity early in exploration to identify promising materials before detailed testing (Coelho et al., 2020).

Another key application is comparative analysis. Because all samples are subjected to identical conditions—particle size, agitation, reagent concentration—the relative performance between ores can be reliably assessed. This is especially useful in geometallurgical programs, where variability between ore domains must be quantified (Pereira, 2019).

Bottle roll testing effectively evaluates grinding impact by varying particle size (e.g., P80 from 75 μm to 300 μm), revealing the link among liberation, extraction, and the energy-metallurgical performance trade-off for process optimization.

In addition, bottle roll tests are well-suited for sensitivity analysis. Parameters such as temperature, pH, acid concentration, and oxidant dosage can be varied independently, allowing controlled assessment of their influence on leaching kinetics. Typical temperature ranges (25–90 °C) and pH conditions (<1.5 in acidic systems) can be explored efficiently, generating valuable data for process understanding.

The appropriate applications of bottle roll testing are summarized in Table 8.

Table 8. Appropriate uses of bottle roll testing in leaching studies. Adapted from Dehaine et al. (2021) and Pereira (2019).

Application	Objective	Typical Output	Technical Value
Rapid screening	Identify reactive ores	Extraction (%) in 24–72 h	Early-stage decision support
Comparative testing	Rank ore types	Relative recovery trends	Geometallurgical mapping
Grinding evaluation	Assess liberation effect	Recovery vs particle size	Optimization of comminution
Sensitivity analysis	Evaluate process variables	Response to pH, T, reagent	Process understanding
Maximum potential estimation	Upper-bound recovery	Peak extraction under ideal conditions	Benchmark for process limits

Table 8 shows that bottle roll testing is most effective as a screening and comparative tool rather than a predictive design method. Its ability to isolate variables and generate rapid results makes it particularly useful in early-stage evaluation and process development.

Critical interpretation

Bottle roll testing defines the maximum leaching performance under ideal conditions, serving as a benchmark rather than a direct prediction.

Misuse occurs when bottle roll data are extrapolated beyond this scope. Without accounting for particle-size effects, mass-transfer limitations, and secondary reactions, the results can lead to overly optimistic expectations of process performance.

Engineering implications

When used appropriately, bottle roll tests support:

- Rapid decision-making during exploration and feasibility stages
- Optimization of grinding strategy based on recovery vs energy trade-offs
- Identification of key process variables (pH, temperature, reagent dosage)
- Establishment of theoretical recovery limits

However, these results must always be complemented with column testing, permeability analysis, and scale-relevant experiments before process design.

While bottle roll testing has clear advantages in controlled laboratory applications, its limitations become evident when used outside its intended scope. Understanding when this method is inadequate is essential to avoid misinterpretation and scale-up failure.

12. Diagnostic versus Predictive Use of Bottle Roll Tests and Operational Decision Framework

Bottle roll tests are widely used in hydrometallurgical studies, but their role is frequently misinterpreted. A critical distinction must be made between their diagnostic and predictive capabilities, as these correspond to fundamentally different levels of process interpretation.

From a diagnostic perspective, bottle roll tests are highly effective. They provide controlled conditions that enable evaluation of intrinsic reactivity, sensitivity to process variables (e.g., pH, temperature, reagent concentration), and comparative behavior among ore types. Under these conditions, the method isolates chemical effects and provides reproducible kinetic trends.

However, their predictive capability is inherently limited. Industrial leaching systems are governed by coupled phenomena—including mass transfer, hydrodynamics, mineralogical heterogeneity, and time-dependent processes such as reprecipitation and permeability loss—that are not captured under bottle-roll conditions. As a result, direct extrapolation of laboratory data leads to systematic bias in the estimation of recovery, acid consumption, and residence time.

The distinction between diagnostic and predictive use is therefore essential to avoid misinterpretation. Bottle roll tests should be treated as tools for screening and mechanistic understanding, rather than as standalone predictors of industrial performance.

To operationalize this distinction, a decision-oriented framework is proposed that links experimental observations to appropriate next steps in process evaluation.

The distinction between diagnostic and predictive use of bottle roll tests can be further clarified through an operational framework that links experimental observations to decision-making criteria. While previous sections have discussed these limitations conceptually, a structured approach is required to guide their practical application in process development and scale-up.

The framework proposed in Figure 8 translates the interpretation of bottle-roll results into a decision-oriented sequence, defining when laboratory data are sufficient and when progression to column-, pilot-, or industrial-scale testing becomes necessary.

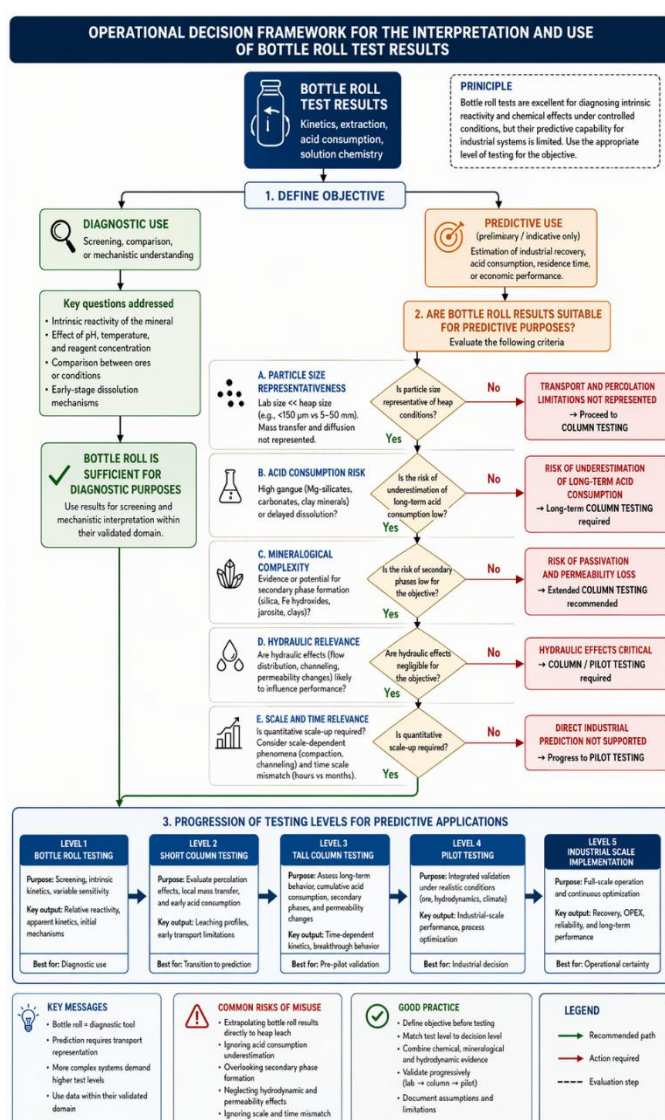


Figure 8. Operational decision framework for the interpretation and use of bottle roll test results. The diagram distinguishes between diagnostic and predictive uses and defines criteria for progression to column-, pilot-, and industrial-scale testing. Prepared by the authors based on a critical synthesis of the reviewed literature.

The proposed framework introduces a structured decision sequence to guide the interpretation of bottle roll results.

The first stage evaluates whether the test objective is diagnostic or predictive. If the objective is diagnostic—such as screening ore reactivity, comparing samples, or assessing sensitivity to operating variables—bottle roll testing is sufficient and provides reliable information.

If predictive use is intended, additional criteria must be evaluated:

- a. **Granulometric discrepancy:** If laboratory particle size differs significantly from heap conditions (e.g., $<150\ \mu\text{m}$ vs 5–50 mm), transport limitations are not represented, and column testing becomes necessary.
- b. **Acid consumption uncertainty:** If acid demand is high or mineralogy suggests delayed reactions (e.g., Mg-silicates), long-term column tests are required to capture cumulative consumption.
- c. **Mineralogical complexity:** The presence of secondary phase formation (e.g., silica gel, iron hydroxides, jarosite) indicates risk of passivation and permeability loss, requiring extended testing.
- d. **Hydraulic relevance:** If flow distribution, permeability, or irrigation behavior are relevant for process design, bottle roll data alone are insufficient.
- e. **Scale-up objective:** Any estimation of industrial recovery, residence time, or economic performance requires validation beyond laboratory testing.

Based on these criteria, the framework defines a progression:

1. Bottle roll testing → screening and intrinsic kinetics
2. Short column testing → initial transport and percolation effects
3. Tall column testing → time-dependent behavior and scale representation
4. Pilot testing → integrated validation under realistic conditions

This progression reflects the transition from reaction-controlled systems to transport-limited systems and ensures that experimental data are used within their appropriate domain.

13. When Bottle Roll is Inadequate

Bottle roll testing becomes inadequate when used beyond its intended scope as a screening and comparative tool. Its fundamental limitations—fine particle size, homogeneous mixing, and the absence of hydraulic constraints—prevent it from capturing key phenomena

that govern industrial leaching systems. When applied to design or predictive purposes, these limitations can lead to systematic errors in process evaluation.

Bottle roll tests are not suitable for heap leach design. Industrial heaps operate under percolation-driven flow, where particle size distribution (typically 5–50 mm), pore structure, and solution pathways determine performance. In contrast, bottle roll tests eliminate these constraints by employing finely ground material and continuous agitation, thereby yielding artificially enhanced kinetics and uniform reagent distribution.

Similarly, bottle roll tests cannot predict hydraulic behavior. Parameters such as permeability, solution distribution, channeling, and drainage are central to heap performance but are completely absent under laboratory bottle-roll conditions. In industrial systems, permeability losses of 20–60% over time are commonly observed due to compaction and secondary phase formation, which directly affect recovery and leaching times.

Estimating acid use from bottle-roll data is unreliable because lab conditions mask delayed mechanisms such as reprecipitation and gangue dissolution. Industrial acid consumption can be 1.5 to 3 times higher, especially in silicate-rich or lateritic ores (Kalbe et al., 2024).

Finally, bottle roll tests are inadequate for evaluating hydraulic stability. Heap systems are subject to dynamic structural changes, including compaction, fines migration, and pore blockage by secondary phases. These effects influence long-term operation and cannot be captured in short-duration, well-mixed laboratory tests.

The main limitations of bottle roll testing when applied to industrial design and prediction are summarized in Table 9.

Table 9. Limitations of bottle roll testing for industrial leaching applications. Adapted from Fernando et al. (2020).

Application	Bottle Roll Capability	Industrial Requirement	Limitation
Heap design	Not representative	Particle size 5–50 mm, percolation flow	Overestimates kinetics and recovery
Hydraulic prediction	Not applicable	Permeability, flow distribution	No simulation of fluid flow
Acid consumption estimation	Underestimated	Time-dependent, gradient-driven	Ignores secondary reactions
Hydraulic stability	Not captured	Long-term structural evolution	No compaction or pore blocking

Table 9 shows that bottle roll testing does not replicate key physical and chemical constraints of industrial leaching, causing it to overestimate performance and underestimate operational challenges.

Critical interpretation

The central issue is the mismatch between laboratory conditions and industrial reality. Bottle roll tests eliminate the effects of mass-transfer limitations, hydraulic behavior, and structural evolution of the ore bed. These factors, however, dominate large-scale operations.

As a consequence, relying on bottle roll data for design decisions can result in unrealistic expectations during project development. Common outcomes include longer-than-expected leaching cycles, higher reagent consumption, and reduced metal recovery during operation.

Engineering implications

Using bottle roll results outside their appropriate scope can lead to:

- a. Incorrect sizing of leach pads and irrigation systems
- b. Underestimation of acid supply and storage requirements
- c. Poor prediction of recovery and project economics
- d. Operational instability due to unaccounted hydraulic effects

To avoid these issues, bottle roll testing must be complemented with:

- a. Column leach tests under controlled flow conditions
- b. Permeability and hydraulic characterization
- c. Long-term kinetic monitoring
- d. Integrated geometallurgical modeling

Given these limitations, it is essential to establish clear guidelines for the correct use of bottle roll testing within a broader experimental and design framework.

Understanding bottle roll testing involves recognizing its suitable uses and limitations. It offers quick insights into leaching but is often misused in design and scale-up. See Figure 9 for clarification.

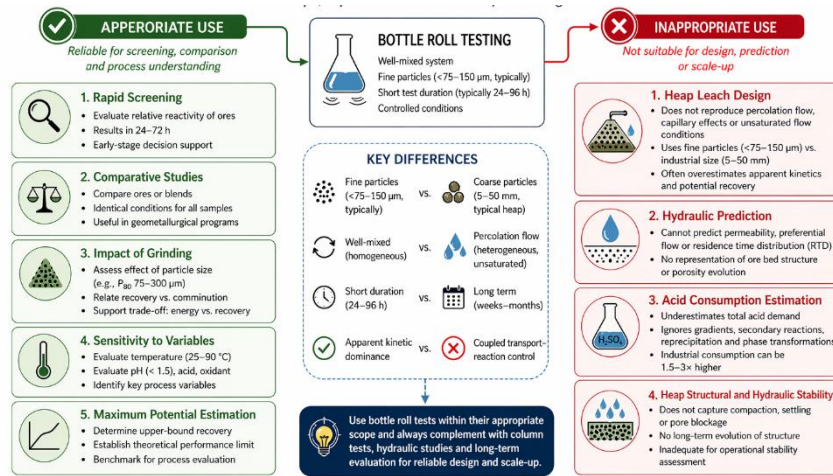


Figure 9. Adequate vs. inadequate use of bottle roll testing in leaching studies. The figure contrasts reliable applications (screening, comparison, and sensitivity analysis) with common misuses related to design, scale-up, and prediction, highlighting the underlying physical and operational differences between laboratory and industrial systems. Adapted from Guner et al. (2023), Pereira (2019), and Fernando et al. (2020).

Figure 9 highlights that bottle roll testing is most effective when used for comparative and exploratory purposes under controlled conditions. Its ability to isolate variables and generate rapid results makes it suitable for screening and sensitivity analysis. However, the figure also emphasizes that these advantages become limitations when the method is extrapolated to industrial systems.

In particular, the absence of hydraulic behavior, coarse particle effects, and long-term reactions leads to systematic errors in predicting heap performance. Misinterpretations, such as equating rapid extraction with process viability or laboratory acid consumption with industrial demand, can lead to flawed design decisions. Therefore, bottle roll testing should be positioned as a preliminary tool within a broader experimental framework that includes column testing, hydraulic evaluation, and geometallurgical integration.

14. Recommended Best Practices

The reliability of bottle roll testing depends not only on experimental execution but also on the quality and completeness of data reporting. Inconsistent or incomplete reporting remains a major limitation in the literature, often preventing reproducibility and limiting the value of results for process evaluation and scale-up.

To ensure technical robustness, a minimum set of parameters must be systematically reported. These parameters define the chemical environment, reaction kinetics, and boundary conditions of the test, and are essential for interpreting results in a broader process context.

The minimum recommended dataset for bottle roll testing is summarized in Table 10.

Table 10. Minimum reporting requirements for bottle roll testing. Adapted from Pereira (2014).

Parameter	Unit	Typical Range	Technical Importance
Particle size distribution (PSD)	µm / mm	<150 µm (lab)	Controls surface area and liberation
Solid-to-liquid ratio (S/L)	% w/w	10–50%	Influences mass transfer and reagent availability
Initial acid concentration	g/L H ₂ SO ₄	5–50 g/L	Defines leaching environment
Final acid concentration	g/L H ₂ SO ₄	Variable	Indicates acid consumption
Temperature	°C	20–90 °C	Affects reaction kinetics
pH profile (vs time)	–	<1.5 (acidic systems)	Reflects reaction progress
Eh profile (vs time)	mV	400–800 mV	Indicates redox conditions
Leaching time	h	24–72 h	Defines kinetic window
Residue characterization	–	Mineralogical/chemical	Identifies unreacted phases and passivation

Table 10 highlights that bottle roll testing should not be limited to reporting final extraction values. Time-resolved data, particularly pH and Eh evolution, are critical for understanding reaction mechanisms and identifying potential secondary processes such as reprecipitation or passivation.

Critical interpretation

A major weakness in many studies is the absence of dynamic process data. Reporting only initial and final conditions obscures important transitions in system chemistry, including acid depletion, redox shifts, and secondary phase formation. Without these data, it is not possible to assess whether observed kinetics are controlled by reaction rate, diffusion, or changes in chemical environment.

In addition, post-test residue characterization is often neglected. Mineralogical analysis (e.g., XRD, SEM-EDS) provides direct evidence of incomplete liberation, surface passivation, or formation of secondary phases, all of which are critical for interpreting leaching performance.

A robust evaluation of leaching performance requires the integration of laboratory and scale-representative testing methods. Bottle roll tests provide rapid kinetic insights under controlled conditions, whereas column tests capture transport limitations and hydraulic behavior. The combined workflow from screening to process design is outlined in Figure 10.

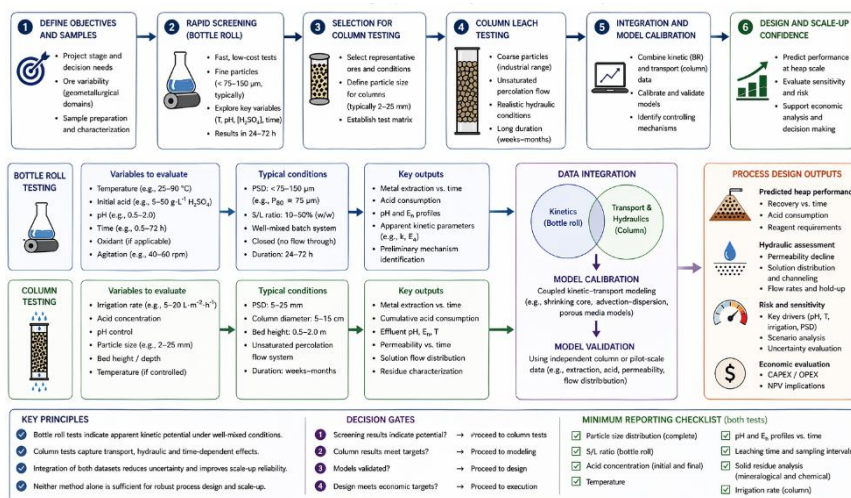


Figure 10. Recommended workflow integrating bottle roll and column leach testing for reliable leaching evaluation and scale-up. The figure illustrates the sequential use of bottle roll tests for screening and sensitivity analysis, followed by column tests for transport and hydraulic validation, culminating in model calibration and process design. Adapted from Pereira (2014), Walder et al. (2022), and Lin et al. (2023)

Figure 10 demonstrates that bottle roll and column tests provide complementary information rather than interchangeable results. Bottle roll testing defines the upper-bound kinetic response under ideal mixing conditions, whereas column testing introduces key industrial constraints, including particle-size distribution, percolation flow, and time-dependent behavior.

The integration step is crucial. Kinetic data from bottle roll tests must combine with transport and hydraulic data from column experiments for accurate model calibration. Without this, recovery, acid use, and residence time predictions are unreliable.

In practical terms, the workflow reduces uncertainty during scale-up by introducing validation stages before process design. Decision gates—such as confirmation of recovery targets and model validation—ensure that only technically robust datasets are used for engineering calculations and economic evaluation.

15. Integration with Column Testing

Reliable scale-up of leaching systems requires combining lab kinetic data with transport and hydraulic constraints typical of industrial settings, which bottle roll tests alone can't do. A structured framework blending bottle roll and column experiments is essential to reduce uncertainty and improve predictions.

The proposed approach follows a sequential methodology, in which each testing stage contributes distinct and complementary information:

1. Bottle roll testing defines intrinsic leaching kinetics under ideal mixing conditions, establishing the upper-bound extraction potential.
2. Preliminary modeling uses bottle roll data to estimate kinetic parameters (e.g., apparent rate constants, activation reaction energy) and identify potential rate-controlling mechanisms.
3. Short-column testing introduces initial hydraulic effects, allowing evaluation of permeability, solution distribution, and early-stage flow behavior under controlled conditions.
4. Tall-column testing simulates industrial conditions more closely, incorporating realistic particle size distributions (typically 5–25 mm), residence times (weeks to months), and vertical gradients.
5. Pilot-scale testing validates process performance under near-industrial conditions, confirming recovery, acid consumption, and operational stability.

The integration of bottle roll and column testing within a structured scale-up framework is illustrated in Figure 11.

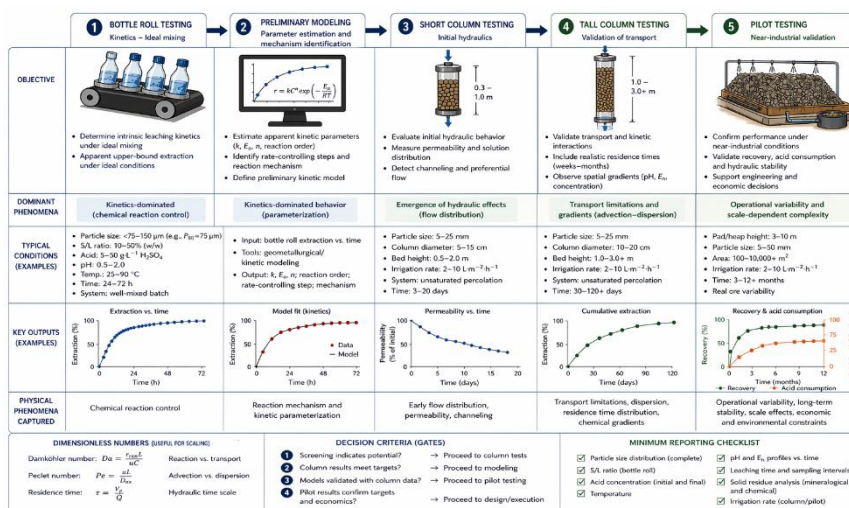


Figure 11. Integrated testing framework for leaching scale-up: bottle roll testing, column experiments, and pilot validation. The figure illustrates the progressive incorporation of kinetic, hydraulic, and transport effects from laboratory to near-industrial conditions. Adapted from Fagan-Endres et al. (2023) and Ortega-Tong et al. (2023).

Figure 11 shows that each testing stage adds physical realism. Bottle roll tests isolate chemical kinetics, whereas column tests incorporate key industrial constraints, including percolation flow, residence time, and mass transfer limitations. Pilot testing further validates

system behavior under operational conditions, including variability in feed material and process control.

Critical interpretation

The central advantage of this integrated approach is the separation and subsequent coupling of kinetic and transport phenomena. Bottle roll tests provide high-resolution kinetic data but eliminate transport limitations. Column tests reintroduce these constraints, allowing identification of the dominant controlling mechanisms under realistic conditions.

Short columns are particularly useful for screening hydraulic behavior and identifying potential issues such as channeling or early permeability loss. Tall columns extend this analysis over longer time scales, capturing delayed effects such as secondary phase formation, reprecipitation, and structural changes in the ore bed.

Without this progression, scale-up models remain incomplete. Direct extrapolation from bottle roll data typically leads to overestimation of recovery and underestimation of leaching time and reagent consumption.

Quantitative considerations

Typical differences between testing stages include:

- a. Particle size: <150 μm (bottle roll) vs 5–25 mm (columns)
- b. Leaching time: 24–72 h (bottle roll) vs 30–120 days (columns)
- c. Flow regime: fully mixed vs percolation-controlled
- d. Acid consumption: baseline vs 1.5–3 \times higher under column conditions
- e. These differences highlight the need for calibration between datasets before process design.

Engineering implications

An integrated testing workflow enables:

- a. Calibration of kinetic–transport models
- b. Validation of acid consumption and recovery trends
- c. Assessment of permeability and hydraulic stability
- d. Reduction of scale-up risk and uncertainty

From an economic perspective, this approach improves the reliability of OPEX and CAPEX estimates, particularly for large-scale heap leaching operations where residence time and reagent consumption dominate costs.

Despite advances in integrated testing methodologies, significant uncertainties remain due to limited data availability and incomplete understanding of complex leaching systems.

16. Data Gaps and Research Needs

Despite extensive bottle-roll and column testing in leaching studies, significant data gaps remain, limiting scale-up and predictive modeling. These gaps are both experimental and conceptual, especially in integrating kinetics, transport phenomena, and mineralogical controls.

One of the most critical limitations is the scarcity of coupled kinetic–transport models. Most studies treat leaching kinetics independently from mass transfer and hydraulic behavior, even though industrial systems are governed by their interaction. As a result, model predictions often fail when applied beyond laboratory conditions.

Another major gap is the limited integration with quantitative mineralogy. While mineralogical characterization is widely recognized as essential, it is rarely incorporated into kinetic modeling frameworks. The relationship between mineral phase distribution, liberation, and leaching response remains insufficiently quantified (Jia et al., 2024).

A limitation is the lack of systematic correlation between bottle roll and column test results. While both are common, few studies provide direct comparisons under controlled conditions. This prevents developing reliable scaling factors between laboratory and semi-industrial data.

In addition, long-term datasets remain scarce. Most bottle roll tests are limited to 24–72 hours, and even column tests are often restricted to relatively short durations. However, industrial leaching processes frequently operate over periods exceeding 100 days. This mismatch limits the ability to capture delayed phenomena such as secondary phase formation, permeability loss, and progressive acid consumption.

The main data gaps and research needs identified in current leaching studies are summarized in Table 11.

Table 11. Key data gaps and research needs in leaching studies. Adapted from Jia et al. (2024).

Area	Data Gap	Technical Impact	Research Need
Kinetic modeling	Limited coupling with transport	Poor scale-up prediction	Develop coupled kinetic–transport models
Mineralogy integration	Limited quantitative linkage	Uncertain mechanism identification	Integrate QEMSCAN/MLA with kinetics
Bottle roll vs column correlation	Scarce comparative datasets	No reliable scaling factors	Generate paired datasets under controlled conditions

Long-term behavior	Lack of >100 day data	Incomplete understanding of delayed effects	Conduct extended column and pilot tests
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Table 11 shows that current limitations are interconnected. The absence of long-term data, for example, directly affects the development of coupled models and the understanding of mineralogical effects. Similarly, the lack of correlation between test methods prevents the validation of predictive frameworks.

Critical interpretation

The central issue is not the lack of individual datasets, but the absence of integrated datasets that combine kinetics, transport, and mineralogical information over relevant time scales. Without this integration, models remain empirical and site-specific, limiting their applicability to new deposits or operating conditions.

This gap also affects economic evaluation. Uncertainty in recovery, acid consumption, and residence time propagates directly into OPEX and CAPEX estimates, increasing project risk and reducing confidence in feasibility studies.

Engineering implications

From a process design perspective, these data gaps result in:

- a. High uncertainty in scale-up predictions
- b. Conservative or inaccurate design margins
- c. Increased need for pilot testing
- d. Limited transferability of results between projects

Addressing these gaps requires coordinated experimental and modeling efforts, including standardized reporting protocols, long-term testing campaigns, and integration of advanced characterization techniques.

To overcome these limitations, a structured conceptual framework is required to integrate experimental data, modeling approaches, and process design considerations.

17. Proposed Conceptual Framework

A consistent interpretation of bottle roll testing requires a clear conceptual separation between intrinsic chemical kinetics and the multi-physics processes governing industrial heap leaching. Bottle roll tests provide valuable information, but only within a limited domain. A unified framework is therefore necessary to correctly position these data within the broader context of process performance.

Bottle roll testing primarily measures intrinsic chemical kinetics under idealized conditions. Fine particle size, homogeneous mixing, and absence of flow constraints isolate the reaction rate at the mineral–solution interface. This represents the upper-bound kinetic response of the system.

In contrast, heap leaching performance depends on the interaction of multiple coupled processes. These include:

- Chemical kinetics, governing dissolution reactions
- Mass transfer, controlling diffusion within particles and through pore networks
- Hydrodynamics, defining solution distribution and flow pathways
- Phase transformations, including precipitation and reprecipitation
- Mechanical effects, such as compaction and structural evolution of the heap

The relative importance of these factors varies with time and operating conditions. Early stages may be dominated by reaction kinetics, while transport limitations and structural changes in the ore bed often control long-term performance.

The relationship between intrinsic kinetics and industrial leaching performance can be conceptualized as a hierarchy of controlling mechanisms, as illustrated in Figure 12.

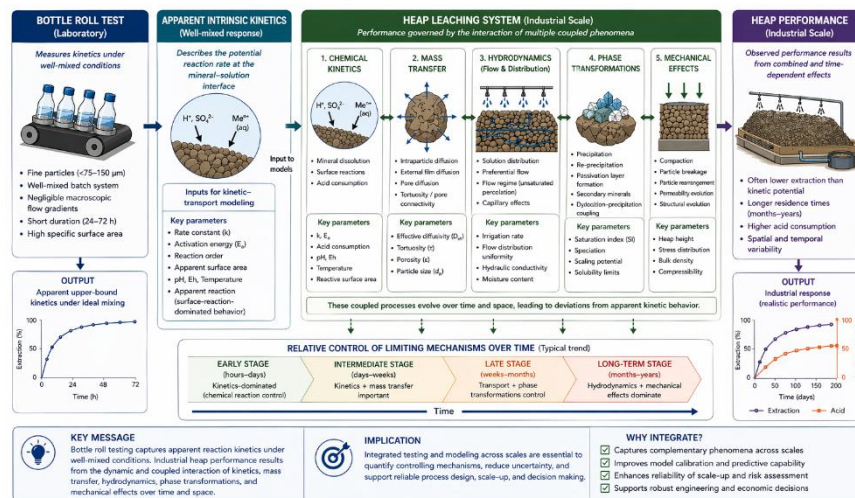


Figure 12. Conceptual framework linking intrinsic kinetics and heap leaching performance. Bottle roll testing captures intrinsic chemical kinetics, whereas industrial performance depends on the interplay among kinetics, mass transfer, hydrodynamics, phase transformations, and mechanical effects. Adapted from Nicol (2022) and León et al. (2025).

Figure 12 illustrates that bottle roll testing represents only one dimension of a multi-factor system. While it provides essential information on reaction potential, it does not account for the constraints that ultimately control industrial performance.

The framework highlights that heap leaching is a coupled system in which limitations shift over time. For example, initial leaching rates may align with bottle-roll predictions, but deviations increase as transport limitations, permeability loss, and secondary-phase formation become dominant. This explains why high extraction values obtained in laboratory tests often fail to translate into equivalent industrial performance.

Critical interpretation

The key implication of this framework is that bottle roll results should be interpreted as kinetic benchmarks rather than predictive outcomes. Their role is to define the maximum achievable reaction rate under ideal conditions, which must then be adjusted based on transport and hydraulic constraints.

Misinterpretation arises when this distinction is ignored. Treating bottle roll data as directly scalable leads to systematic overestimation of recovery and underestimation of leaching time and reagent consumption.

Engineering implications

From a design perspective, the framework supports a shift from single-parameter evaluation to integrated process analysis. Reliable prediction of heap performance requires:

- a. Coupling kinetic models with mass transfer and flow models
- b. Incorporating mineralogical constraints into reaction frameworks
- c. Accounting for time-dependent phenomena such as precipitation and compaction
- d. Validating predictions through column and pilot testing

This integrated approach improves the accuracy of recovery forecasts, acid consumption estimates, and economic evaluations (OPEX and CAPEX).

The proposed conceptual framework provides a structured basis for interpreting bottle roll data and integrating it into industrial process design. It emphasizes the need for multi-scale analysis and highlights the risks associated with oversimplified interpretations.

18. Conclusion

Bottle roll testing remains one of the most widely used methods for evaluating leaching performance. Its simplicity, low cost, and rapid execution make it an effective tool for early-stage screening, comparative analysis, and sensitivity studies. When properly applied, it provides valuable insight into intrinsic chemical kinetics and defines the upper-bound extraction potential of a given system.

However, this review demonstrates that bottle roll results are frequently misinterpreted when extrapolated to industrial conditions. The method isolates chemical kinetics under idealized conditions, but industrial heap leaching is governed by the interaction of multiple coupled phenomena, including mass transfer, hydrodynamics, phase transformations, and mechanical effects. As a result, bottle roll tests systematically overestimate extraction rates and underestimate leaching time and reagent consumption.

Key discrepancies identified in this work include the effects of particle-size reduction, the absence of hydraulic constraints, the limited representation of secondary-phase formation, and the lack of long-term data. These factors lead to common scale-up errors, particularly in predicting recovery, acid consumption, and operational stability.

To address these limitations, a structured and integrated testing approach is required. The combination of bottle roll and column testing, supported by mineralogical characterization and coupled kinetic–transport modeling, provides a more reliable basis for process evaluation. This approach reduces uncertainty, improves confidence in scale-up, and enhances the accuracy of economic assessments.

From an engineering perspective, bottle roll testing should be positioned as a screening and benchmarking tool, not a predictive design method. Its results must be interpreted within a broader framework that accounts for the physical and chemical constraints of industrial systems.

Future progress in leaching research will depend on the development of integrated datasets, long-term experimental campaigns, and models that explicitly couple kinetics with transport and mineralogical factors. Addressing these gaps is essential to improve predictive capability and reduce risk in industrial leaching operations.

From an economic standpoint, the misuse of bottle roll data can lead to systematic underestimation of both OPEX and CAPEX. In particular, acid consumption and residence time are often misrepresented, resulting in unrealistic cost projections and increased financial risk. Integrating kinetic, transport, and economic considerations is therefore essential for reliable process design and scale-up.

Declarations

Funding

The author received no specific funding for this work.

Conflicts of Interest

The author declares no conflict of interest.

Data Availability

The datasets generated and/or analyzed during the current study are available from the corresponding author on reasonable request.

Author Contributions

Antonio Clareti Pereira: Conceptualization, methodology, investigation, data analysis, writing—original draft preparation, writing—review and editing.

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