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Literature Review: Methods for Nickel Recovery from Spent Electrolytes in Copper Electrorefining

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Abstract: Spent electrolytes generated during copper electrorefining contain increasingly high concentrations of dissolved nickel, which negatively affects cathode quality, decreases current efficiency, and requires regular electrolyte purification. At the same time, nickel is a valuable strategic metal used in stainless steel production, electroplating, and energy-storage applications, making its recovery both economically and environmentally important. This review summarizes recent advances in nickel recovery technologies from spent copper electrorefining electrolytes, focusing on electrochemical, hydrometallurgical, and hybrid approaches. Electrochemical methods such as selective electrodeposition and electrowinning enable the direct recovery of metallic nickel, achieving high purity and moderate energy consumption. Emerging systems based on deep eutectic solvents offer improved selectivity and extended electrochemical windows, representing a promising alternative to traditional aqueous electrolytes. Hydrometallurgical techniques—including solvent extraction, ion exchange, and controlled precipitation—provide high separation efficiency, especially for Cu–Ni systems with complex impurity compositions. Integrated process flows combining copper removal, nickel concentration, and final purification have demonstrated superior performance compared to single-stage methods. Studies on anode slime processing further highlight the potential of pressure leaching for near-complete nickel extraction under optimized acidic and oxidizing conditions. Overall, the literature indicates that well-designed hybrid systems offer the most effective balance of selectivity, energy efficiency, and environmental sustainability for nickel recovery.

Keywords— nickel recovery, spent electrolytes, copper electrorefining, electrochemical methods, solvent extraction, hydrometallurgical processes

1. INTRODUCTION

Copper electrorefining is one of the most efficient industrial processes for producing high-purity copper cathodes for various applications in electrical and electronic industries. During this electrochemical process, copper anodes containing impurities dissolve in sulfuric acid-based electrolytes, while pure copper deposits on the cathode. However, impurities including nickel, which exhibits lower electrode potential than copper, dissolve into the electrolyte rather than forming anode slimes, creating spent electrolytes with significant nickel concentrations [1,8]. The presence of nickel in copper electrorefining systems presents both environmental and economic challenges. Nickel contamination can negatively affect cathode quality, reduce current efficiency, and necessitate regular electrolyte regeneration [2].

The recovery and removal of nickel from spent copper electrorefining electrolytes has become increasingly important due to several factors: the growing emphasis on circular economy principles, the increasing concentration of nickel in recycled copper anodes from electronic waste, and the economic value of nickel as a strategic metal used in stainless steel production and battery technologies. Current literature indicates that nickel concentrations in spent copper refinery electrolytes can range significantly, from approximately 80 to 27,600 µg/g depending on the source material and operational conditions [8].

Over the past decade, significant advances have been made in developing methods for nickel recovery from spent electrolytes. These methods encompass electrochemical, hydrometallurgical, and hybrid approaches. This literature review synthesizes recent research findings from leading international scientific studies to provide a comprehensive analysis of the current state of nickel recovery technologies from spent copper electrorefining electrolytes.

2. MATERIALS AND METHODS

The literature review was carried out through a systematic and structured approach designed to identify, evaluate, and synthesize research on nickel recovery from spent electrolytes produced during copper electrorefining operations. The methodology integrated a comprehensive analysis of global research efforts, reference tracking, and critical assessment of peer-reviewed scientific publications to ensure accuracy and thematic completeness.

Relevant studies were identified through targeted keyword combinations and logical operators to ensure precision and inclusiveness. The primary focus areas included *nickel recovery*, *copper electrorefining*, *spent electrolyte treatment*, *electrowinning*, *solvent extraction*, *ion exchange*, *hydrometallurgical recovery*, and *selective electrodeposition*. To ensure comprehensive coverage, synonymous and contextually related terms were also considered, allowing inclusion of both experimental and review-based studies from diverse international sources.

Publications were included if they reported experimental, pilot-scale, or review data addressing nickel recovery, removal, or separation from copper refinery electrolytes or similar mixed Cu–Ni sulfate systems. Both hydrometallurgical and electrochemical processes were examined, encompassing selective electrodeposition, solvent extraction, ion exchange, precipitation, and integrated hybrid techniques. Conference papers, journal articles, and technical reports were reviewed when they provided sufficient methodological details and quantitative results.

From each selected study, key information was extracted, including:

- recovery mechanism and process type;
- main operating parameters (e.g., temperature, pH, current density, extractant concentration);
- recovery efficiency and nickel purity;
- energy consumption, reagent requirements, and environmental indicators;
- and reported techno-economic or industrial feasibility.

The extracted data were systematically analyzed and compared to identify major trends in process efficiency, selectivity, and sustainability. Special attention was given to innovations involving deep eutectic solvents (DES)**,** hybrid hydrometallurgical–electrochemical systems**,** and environmentally benign separation technologies**.** Collectively, the reviewed literature provides a critical and comprehensive overview of the evolution, challenges, and current advancements in nickel recovery technologies from spent copper electrorefining electrolytes.

3. RESULTS

3.1 Electrochemical Methods

3.1.1 Selective Electrodeposition

Electrodeposition and electrowinning remain among the most significant methods for nickel recovery from spent copper electrorefining electrolytes due to their direct metallurgical output and integration potential with existing industrial processes. Recent research has demonstrated that selective nickel electrodeposition can be achieved from mixed copper-nickel sulfate solutions through careful control of electrochemical parameters [4-6].

The fundamental principle underlying nickel recovery by electrodeposition relies on the difference in standard electrode potentials between copper ($E° = +0.34$ V vs SHE) and nickel ($E° = -0.25$ V vs SHE). Under acidic conditions in traditional sulfate systems, copper preferentially deposits before nickel, enabling potential-dependent separation [6]. However, complete separation of nickel requires modification of standard electrorefining electrolytes.

Co-electrodeposition studies in spouted electrochemical reactors demonstrated that the behavior of copper and nickel deposition from mixed solutions differs significantly from individual metal solutions [7]. The metal displacement reaction between deposited nickel and copper ions in solution effectively reduces copper corrosion while amplifying nickel corrosion. Current efficiency for nickel electrowinning from sulfate electrolytes typically ranges from 60-74%, depending on extraction parameters [18]. Energy consumption for nickel electrodeposition has been reported in the range of 1.82-2.24 kWh/lb of nickel recovered, with variations based on nickel concentration, extraction rate, and temperature [17].

A comprehensive study on nickel recovery from spent electroplating solutions utilizing hydrometallurgical precipitation followed by electrowinning achieved nickel recovery rates of approximately 61% with 99% purity when operating at a cell voltage of 3.5 V for 24 hours [3]. This process involved initial precipitation of nickel hydroxide using sodium hydroxide at pH ≥13, followed by leaching in sulfuric acid at optimal conditions (2 M H_2SO_4, 100:1 g/L solid-to-liquid ratio, 60 minutes), and final electrowinning [3,4].

3.1.2 Deep Eutectic Solvents (DES)

Recent advances in electrochemical nickel recovery employ deep eutectic solvents (DES) as novel electrolyte systems. Gharib et al. (2023) investigated nickel electrodeposition in 1:2 choline chloride: ethylene glycol (Ethaline) at 90°C in the presence of copper ions. The study revealed that the presence of copper ions actually improved nickel electrodeposition quality by minimizing surface roughness and promoting consistent surface morphology through altered metal speciation and mass transport characteristics compared to aqueous solutions [1].

Deep eutectic solvents offer significant advantages over traditional aqueous sulfate electrolytes: they provide lower volatility, extended electrochemical stability windows, and the ability to achieve superior selectivity through speciation control. The electronegativity of Cu in ChCl-EG DES systems was demonstrated to be stronger than that of Ni, with current peaks occurring at +0.3 V and +0.5 V respectively, enabling improved separation [9].

3.2 Hydrometallurgical Methods

3.2.1 Solvent Extraction

Solvent extraction represents a well-established hydrometallurgical technique for nickel-copper separation from sulfate media. Selective metal extraction from mixed solutions exploits differences in metal-ligand complexation strength and pH-dependent extraction behavior.

It has been demonstrated that copper, nickel, and zinc can be selectively extracted from sulfate solutions using LIX 984N at different equilibrium pH values. Copper extraction was achieved at pH 3.5, while nickel extraction occurred at pH 7.3, enabling sequential recovery. Two-stage counter-current extraction simulations achieved quantitative extraction and recovery of individual metals from loaded organic phases [10].

The selectivity of solvent extraction systems has been further enhanced through synergistic combinations of extractants. Studies utilizing LIX 664N in kerosene achieved selectivity factors for copper extraction from nickel-copper mixtures as high as 6000 at pH 2, with up to 80% nickel extraction from raffinate solutions at pH 9 using 30% LIX

664N. Stripping of copper-loaded organic phases with 180 g/L sulfuric acid yielded 98.5% copper recovery in two-stage operations, with the remaining aqueous phase containing concentrated nickel for subsequent recovery [11].

Alternative extractants including DEHPA and Cyanex 272 have been investigated for nickel-cobalt separation from sulfate solutions, with particular application to impurity removal from high-concentration nickel streams [12]. Capric acid in chloroform proved effective for nickel extraction from sodium sulfate solutions, with metal extraction order determined as Cu > Ni > Co, following classical coordination chemistry principles [20].

Phase separation behavior in synergistic extraction systems represents a critical parameter affecting industrial implementation. Studies of HDNNS/4PC mixtures for copper or nickel extraction revealed that viscosity and excess water uptake in loaded organic phases are the most effective parameters controlling separation rates. Small-angle X-ray scattering analysis confirmed microstructural changes in reverse micelle morphology during extraction processes [12].

3.2.2 Ion Exchange

Ion exchange resins provide selective metal removal from copper refinery electrolytes. Chelating resins with specialized functional groups demonstrate superior selectivity for target elements, allowing impurity removal to very low levels.

A novel approach for copper removal from nickel anodic electrolyte via ion exchange following cupric ion deoxidation achieved optimal reduction conditions at: reducing agent dosage of 4.5 times the theoretical requirement, 0.5-hour reaction time at 40°C, and pH 2.0 [13,21]. Breakthrough capacity studies using orthogonal design analysis revealed that breakthrough capacity decreased with increasing linear flow rate, following the equation $Y = 1.559 - 0.194X + 0.0067X^2$. Height-to-radius ratio and initial copper concentration also significantly influenced breakthrough capacity. Desorption efficiency exceeded 100 g/L $CuCl_2$ concentration using NaCl solution supplemented with 4% (v/v) H_2O_2 [13].

Ion exchange has also been applied to removal of copper, zinc, and nickel from cobalt electrolytes in industrial operations. Cationic exchange resins such as Ceralite IR 120 achieved maximum adsorption capacities of 164 mg/g for Cu(II), 109 mg/g for Ni(II), demonstrating the potential for selective metal purification in complex solutions [15].

3.2.3 Precipitation Methods

Selective precipitation represents a complementary hydrometallurgical technique for nickel recovery from spent electrolytes. Following copper electrowinning or solvent extraction, nickel-containing solutions can undergo controlled precipitation to produce nickel hydroxide or nickel sulfate crystals.

Research on copper and nickel recovery from acidic polymetallic wastewaters demonstrated that copper electrorecovery followed by nickel precipitation through neutralization achieved 99% nickel recovery with purity exceeding 98% as nickel hydroxide. The thermodynamic feasibility of this two-step process was confirmed through theoretical analysis of electrochemical and precipitation reactions in mixed copper-nickel systems [15].

3.3 Anode Slime Processing and Nickel Extraction

Copper anode slimes generated during electrorefining contain valuable metals including silver, gold, platinum group metals, and significant quantities of nickel. While anode slimes are primarily associated with metals having electrode potentials more noble than copper, nickel oxides and nickel-containing compounds in anode slimes represent important secondary nickel sources.

Pressure leaching of nickel-rich anode slimes under oxidized conditions demonstrated maximum nickel extraction of 99.7% using 20% H_2SO_4 at 160°C, 8 bar O_2 pressure, and 200 g/L solid-to-liquid ratio. Under these conditions, copper extraction reached 97-99%, with selenium, tellurium, and arsenic also being recovered. Mineralogical analysis via SEM-EDX revealed the presence of various oxide phases in leach residues [16].

Hydrometallurgical processing of anode slimes indicated that maximum copper recovery of approximately 77% was achievable through hydrochloric acid leaching at 80°C, while nickel recovery remained poor (2.01%) under these conditions. However, applying 20% sulfuric acid concentration with elevated pressure increased nickel extraction substantially, suggesting that high-acid, high-pressure conditions favor nickel recovery from anode slimes containing nickel oxides [14].

3.4 Nickel Contamination in Copper Electrorefining and Process Parameters

Understanding nickel behavior in copper electrorefining electrolytes is essential for optimizing recovery operations. Recent research has characterized nickel contamination mechanisms at the cathode and determined operational parameters affecting nickel accumulation in electrolytes.

Sahlman et al. (2025) demonstrated that increasing Ni^{2+} concentration from 0 to 40 g/L resulted in progressively rougher cathode surfaces and altered morphology of electrodeposited copper. Current efficiency of copper electrorefining decreased with increasing nickel concentration in the electrolyte, attributed to enhanced nodulation and particle entrapment. Electrolyte additives and pH control were found to significantly influence the effects of nickel contamination [2].

Nickel cathode contamination occurs through three primary mechanisms: particle entrapment of nickel oxide anode slime, electrolyte inclusion within cathode deposits, and co-electrodeposition of nickel ions. Experimental investigations using both simulated and industrial anode slimes confirmed that particle entrapment is the major source of cathode nickel contamination, while co-electrodeposition of Ni^{2+} is thermodynamically unlikely under typical copper electrorefining conditions [8].

The effect of antimony, nickel, and sulfuric acid concentration on copper electrorefining revealed that passivation of the anode is more closely related to SO_4^{2-} concentration than Ni concentration, although high nickel levels do decrease current efficiency. Passivation facilitation was observed with increased lead content in anodes, suggesting that slime layer composition and density significantly affect the electrochemical process [8].

4 Discussion

4.1 Comparative Analysis of Nickel Recovery Methods

The literature survey reveals that no single method emerges as universally superior for nickel recovery from spent copper electrorefining electrolytes; rather, method selection depends on specific process requirements, economic considerations, and operational constraints.

Electrochemical methods including electrowinning and electrodeposition offer direct metallurgical output suitable for direct reuse in battery production and stainless-steel manufacturing. Current electrowinning configurations achieve 60-74% current efficiency, requiring careful management of hydrogen ion generation and electrolyte pH [17]. The development of deep eutectic solvent-based electrochemical systems represents a significant advance, potentially enabling superior selectivity and improved energy efficiency compared to traditional aqueous sulfate electrolytes [1,9].

Solvent extraction methods provide high selectivity (up to 6000-fold for copper over nickel) and have been extensively validated in industrial settings [10]. However, solvent extraction introduces organic solvents requiring environmental management and potential recycling, which increases operational complexity and cost. Multi-stage counter-current configurations achieve high recovery rates but require significant capital investment in equipment [10,11].

Ion exchange demonstrates rapid kinetics and exceptional selectivity through specialized chelating resins. However, ion exchange exhibits capacity limitations requiring regular resin regeneration and disposal of spent resin, with environmental implications from chemical regenerants [13].

Precipitation methods are economically attractive for final polishing steps following electrowinning or solvent extraction, achieving high purity products (>98% Ni). However, precipitation alone cannot achieve initial separation from copper in concentrated sulfate media [15].

4.2 Technical Advances and Process Integration

Recent literature indicates significant progress in process integration and hybrid methodologies combining complementary techniques. Sequential processes (electrowinning for copper removal followed by solvent extraction or precipitation for nickel concentration) have emerged as practical industrial approaches [6,15].

The application of deep eutectic solvents in electrochemical nickel-copper separation represents a notable technical advance with potential for industrial implementation. DES systems offer advantages of non-flammability, extended electrochemical stability, lower volatility, and improved metal speciation compared to aqueous media [1,9]. However, higher operating temperatures (80-90°C) and specialized equipment requirements may present implementation challenges.

Advanced electrochemical techniques including the use of charged polymer interfaces to modulate electrodeposition selectivity have been demonstrated for cobalt-nickel separation. While not yet reported specifically for copper-nickel separation, these polymer-modified interfaces represent emerging technology with potential applications to nickel recovery from spent copper electrolytes [5].

4.3 Energy Consumption and Economic Viability

Energy consumption represents a critical factor in economic viability of nickel recovery operations. Literature values indicate:

Electrowinning: 1.82-2.24 kWh/lb Ni recovered [17]

Selective electrodeposition in alternative media (e.g., DES): potentially 18% more energy efficient than conventional methods [19]

Solvent extraction coupled with electrowinning: variable depending on configuration, but substantial electrical energy requirements for electrowinning component

The economic value of recovered nickel, currently trading at $7-9 per pound (market-dependent), must be weighed against recovery operation costs including energy, chemical reagents, equipment maintenance, and personnel.

4.4 Environmental Considerations

Copper electrorefining spent electrolytes represent hazardous waste streams due to nickel content and high acid concentrations. Effective nickel recovery reduces environmental contamination risk and supports circular economy principles. Hydrometallurgical methods (solvent extraction, precipitation) generate potentially recyclable byproducts but require careful management of organic solvents and process liquors [6,10,11].

Life cycle assessment data for analogous recovery processes indicate that hydrometallurgical approaches can achieve negative global warming potential values, making them environmentally favorable compared to pyrometallurgical alternatives [18].

5 CONCLUSION

In recent years, substantial progress has been achieved in developing methods for nickel recovery from spent copper electrorefining electrolytes. Electrochemical methods including selective electrodeposition and electrowinning provide direct metal recovery suitable for industrial reuse, with current efficiency values of 60-74% and nickel purity exceeding 99%. Hydrometallurgical approaches utilizing solvent extraction (achieving selectivity factors >6000) and ion exchange demonstrate high selectivity and economic potential for integrated operations. Deep eutectic solvent-based electrochemical systems represent emerging technology with significant promise for improved selectivity and energy efficiency.

The choice of recovery method should be determined by specific operational requirements including nickel concentration in spent electrolytes, required purity of recovered nickel, capital and operational cost constraints, and environmental regulations. Hybrid methodologies combining complementary techniques (e.g., electrowinning followed by solvent extraction and precipitation) show considerable promise for achieving optimal techno-economic performance while supporting circular economy objectives.

Future research should focus on: (1) development of novel solvents and electrochemical media enabling enhanced selectivity and reduced energy consumption, (2) comprehensive life cycle and techno-economic analyses comparing integrated recovery systems, (3) scaling of deep eutectic solvent-based processes to industrial capacity, and (4) investigation of combined recovery of multiple impurities (As, Sb, Bi, Se, Te) alongside nickel from spent copper refinery streams.

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