

Evaluation of Nickel Removal Efficiency from Wastewater by Electrochemical Method Using Graphite–Stainless Steel Electrodes

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ABSTRACT

This study evaluated the efficiency of nickel ion (Ni^{2+}) removal from electroplating wastewater using a laboratory-scale electrochemical system equipped with a non-sacrificial graphite anode and a grade 304 stainless steel cathode. The primary objective was to investigate the influence of key operational parameters, including initial solution pH (7.0-9.0), applied voltage (5.0-15.0 V), electrolysis duration (30.0-120.0 min), and initial nickel concentration ($C_0 = 567.00 \text{ mg/L}$) to identify optimal processing conditions, determine specific energy consumption, and evaluate complete nickel mass balances across system phases. Residual nickel concentrations were quantitatively analyzed using Flame Atomic Absorption Spectrometry (F-AAS) according to SMEWW 3111B:2023, while system stability was evaluated by monitoring total dissolved solids (TDS) and electrical conductivity (EC) across triplicate runs ($n = 3$). Experimental results demonstrated that under optimal operational parameters (pH 7.0, applied voltage 10.0 V, and electrolysis duration 60.0 min), the maximum nickel removal efficiency reached 78.94%, reducing the residual concentration to 119.41 mg/L with a low specific energy consumption of 0.285 kWh/m^3 . A neutral medium (pH 7.0) exhibited superior operational stability ($\text{SD} = 2.62$, $\text{RSD} = 3.83 \%$). Applied voltage proved to be a critical determinant for cathodic metal deposition; however, raising the potential to 15.0 V triggered parasitic hydrogen evolution, substantially increasing energy consumption to 0.612 kWh/m^3 with marginal efficiency gains. Comprehensive mass balance calculations confirmed that 91.2% of the removed nickel was recovered directly as solid elemental plating on the cathode, while 8.8% formed loose hydroxide precipitates. Although direct electrolysis avoids primary chemical sludge generation, the remaining residual nickel concentration (119.41 mg/L) exceeds national discharge thresholds (QCVN 40:2025/BTNMT, Column A), highlighting the necessity of combining this process with downstream polishing stages (e.g., ion exchange or nanofiltration). Overall, the graphite–stainless steel system offers a sustainable, low sludge alternative for direct heavy metal recovery in industrial wastewater management.

Keywords: Electrochemistry, electroplating wastewater, graphite–stainless steel electrode, mass balance, nickel removal.

INTRODUCTION

In the era of rapid industrialization and modernization, the electroplating, surface finishing, and metallurgy industries play a pivotal role in economic growth. However, these industrial sectors release substantial volumes of hazardous wastewater containing high concentrations of toxic heavy metals into aquatic ecosystems (Fu & Wang, 2011; Qasem et al., 2021). Among these contaminants, nickel (Ni) is extensively utilized due to its superior corrosion resistance, mechanical strength, and aesthetic plating properties (Barakat, 2011). Consequently, effluents generated from plating operations and rinsing processes consistently contain Ni^{2+} concentrations exceeding the permissible limits set by national environmental regulations, such as QCVN 40:2025/BTNMT (Adeyemi & Adebayo, 2022). The persistence of nickel ions in aquatic environments poses

severe ecological hazards owing to their non-biodegradability and strong tendency to bioaccumulate through food chains, leading to acute and chronic toxicity in aquatic organisms (Brix et al., 2017). Human exposure to excessive nickel levels through contaminated water sources is associated with severe health complications, including dermatological disorders, respiratory distress, hepatic and renal dysfunctions, and carcinogenesis (Das et al., 2008; Genchi et al., 2020).

Conventional treatment methodologies for heavy metal wastewater, such as chemical precipitation, adsorption, ion exchange, and membrane filtration, have been widely implemented with varying degrees of success.

Nevertheless, these conventional techniques suffer from notable technical and economic drawbacks. Chemical precipitation generates secondary pollution in the form of massive toxic metal hydroxide sludge requiring costly disposal (Moussavi et al., 2021), whereas adsorption and membrane filtration entail substantial operational expenditures, frequent media regeneration, and severe membrane fouling (Babel & Kurniawan, 2003; Kurniawan et al., 2006). In contrast, electrochemical technologies offer a highly versatile, chemical-free alternative with compact footprints and low environmental footprints (Brião et al., 2024).

Among electrochemical pathways, electrocoagulation (EC) and electroflotation (EF) are frequently used; EC utilizes sacrificial anodes (such as Fe or Al to produce *in situ* hydroxides ($\text{Fe}(\text{OH})_3$ or $\text{Al}(\text{OH})_3$) that entrap metals (Moussavi et al., 2021), while EF leverages micro-bubbles (H_2 and O_2) generated at electrodes to float precipitated flocs to the surface (Barakat, 2011). However, both processes are constrained by continuous electrode consumption, surface passivation, and substantial hazardous sludge generation. Direct electrolysis overcomes these limitations by using direct electrical current (DC) to drive cathodic electroreduction ($\text{Ni}^{2+} + 2\text{e}^- \rightarrow \text{Ni}$), recovering high purity metallic nickel directly on the cathode surface while minimizing secondary waste (Njau et al., 2000).

While direct electrolysis can exhibit higher specific energy consumption when treating low concentration effluents due to mass transport limitations, optimizing operational variables such as current density, solution pH, applied voltage, and electrode configuration can significantly enhance its economic viability (Njau et al., 2000; Qasem et al., 2021). To address these operational challenges, this study investigates the performance of an electrochemical batch reactor utilizing a cost-effective, low solubility graphite anode paired with a 304 stainless steel cathode for the removal and recovery of Ni^{2+} from electroplating wastewater. The core objective is to systematically evaluate the influence of critical operating parameters specifically initial pH, applied voltage (U), and electrolysis duration (t) to determine optimal processing parameters, establish a complete nickel mass balance, evaluate specific energy consumption (E), and discuss the integration of downstream polishing operations for full compliance with discharge standards.

MATERIALS AND METHODS

Wastewater characteristics and reactor setup

The experimental wastewater was collected from actual electroplating effluent produced by Tinh Cong Industrial Joint Stock Company (Di An Ward, Ho Chi Minh City, Vietnam), supplemented with analytical grade nickel sulfate hexahydrate ($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, Merck) dissolved in double-distilled water to establish a standardized initial concentration ($C_0 = 567.00 \text{ mg/L}$). Sodium hydroxide (NaOH , 1 M) and sulfuric acid (H_2SO_4 , 1 M) were used to adjust the initial solution pH to designated values. Sodium chloride (NaCl) was added as a supporting electrolyte (1.0 g/L) to maintain adequate ionic conductivity across test solutions.

The electrochemical reactor consisted of a cylindrical glass vessel with an effective working volume of 2.0 L. The electrode assembly comprised a high-purity graphite plate (100 mm x 50 mm x 5 mm) functioning as the non-sacrificial anode and a grade 304 stainless steel plate (100 mm x 50 mm x 1.5 mm) acting as the cathode. Both electrodes were arranged in a parallel configuration at a fixed inter-electrode spacing of 2.0 cm. Electric potential was supplied by a regulated direct current (DC) power supply (0-30 V, 0-5 A).

Experimental design, replicates, and statistical controls

To evaluate parameter dependencies, experiments were conducted using a controlled single-factor approach.

The initial solution pH was investigated across values of 7.0, 8.0, and 9.0 at a constant applied voltage of 5.0 V and an electrolysis duration of 60.0 min. The effect of applied voltage was subsequently evaluated at 5.0 V, 10.0 V, and 15.0 V under a constant initial pH of 7.0 for 60.0 min. Finally, the influence of electrolysis duration was systematically tracked at intervals of 30.0, 60.0, and 120.0 min under optimized settings of pH = 7.0 and $U = 10.0$ V. To establish data reliability and ensure statistical rigor, all experimental runs were executed in independent triplicates ($n = 3$), with results reported as mean values $\pm$ standard deviation (SD) and measurement precision verified through relative standard deviation (RSD, %) calculations.

Analytical Methods and Mass Balance Calculations

Physicochemical parameters including pH, electrical conductivity (EC), and total dissolved solids (TDS) were monitored in real time using calibrated digital meters (ARC pH meter and Daimito EC/TDS meter). Samples collected before and after electrolysis were filtered through 0.45 μ m membrane filters and analyzed for residual Ni^{2+} concentration using Flame Atomic Absorption Spectrometry (F-AAS, Shimadzu AA-7000) per standard method SMEWW 3111B:2023. Ni^{2+} removal efficiency (H, %) was calculated according to Equation (1):

$$H = \frac{(C_0 - C_t)}{C_0} \times 100\% \quad (1)$$

where C_0 and C (mg/L) represent the concentrations of Ni^{2+} at the initial state and at time t , respectively.

The specific energy consumption per unit volume (E , kWh/m³) was evaluated using Equation (2):

$$E = \frac{(U \times I \times t)}{(V \times 1000)} \quad (2)$$

where U is the applied potential (V), I is the operating current (A), t is the electrolysis time (hours), and V is the volume of treated wastewater (L).

To track metal partitioning, total removed nickel mass (Δm_{total} , g) was compared against the mass gain of the cathode ($\Delta m_{\text{cathode}}$, g) and anode mass change (Δm_{anode} , g) measured gravimetrically after washing and oven-drying at 105°C for 2 h. Cathodic metal recovery efficiency (R_{cat} , %) was quantified using Equation (3):

$$R_{\text{cat}} = \frac{\Delta m_{\text{cathode}}}{(C_0 - C_t) \times V \times 1000} \times 100\% \quad (3)$$

RESULTS AND DISCUSSION

Effect of initial solution pH on treatment performance and stability

Initial pH strongly dictates electrochemical pathways by altering metal ion speciation, electrode surface charges, and bulk solution conductivity. As initial pH was increased from 7.0 to 9.0, Ni^{2+} removal efficiency dropped progressively from 69.04 % to 56.57 % ($U = 5.0$ V, $t = 60.0$ min) (Fig. 1).

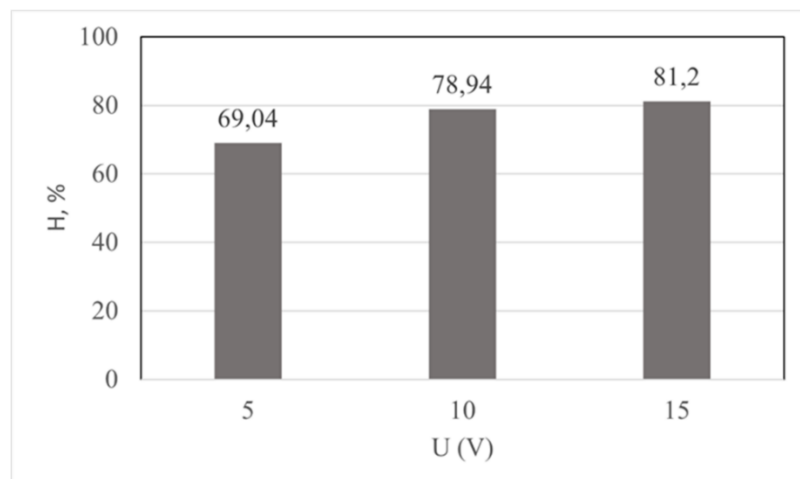


Figure 1. Dynamics of Ni^{2+} removal efficiency across different operating voltages at pH = 7.0, $t = 60$ min.

The decline in efficiency at elevated pH values stems from cathodic passivation. In alkaline environments, local accumulation of hydroxide ions (OH^-) promotes premature precipitation of nickel hydroxide ($\text{Ni}(\text{OH})_2$), forming an insulating layer over the stainless steel cathode. This passive film increases charge-transfer resistance, suppressing direct electroreduction ($\text{Ni}^{2+} + 2e^- \rightarrow \text{Ni}$). Consequently, operating current fell from 0.175 A at pH 7.0 to 0.128 A at pH 9.0. Solution conductivity and TDS reductions followed a matching downward trend. Statistical evaluations confirmed superior reproducibility at pH 7.0 (SD = 2.62, RSD = 3.83 %), establishing neutral pH as optimal (Table 1).

Table 1. Operating parameters and performance indicators under varying pH conditions (U = 5.0 V, t = 60.0 min, n = 3)

Parameter / Indicator	pH = 7.0	pH = 8.0	pH = 9.0
Power supply Voltage - Current	5 V - 0.175 A	5 V - 0.148 A	5 V - 0.128 A
Output Power (W)	0.875	0.74	0.64
Initial Ni^{2+} Concentration C_0 (mg/L)	567	567	567
Residual Ni^{2+} Concentration C_t (mg/L)	175.54 ± 5.12	211.28 ± 3.01	246.27 ± 3.50
Ni^{2+} Removal Efficiency (%)	69.04	62.74	56.57
TDS Reduction Efficiency (%)	38.65	30.07	21.72
EC Reduction Efficiency (%)	38.83	30.53	22.35
Specific Energy Consumption (E, kWh/m ³)	0.146	0.123	0.106
Graphite Anode Mass Loss (g)	0.027	0.035	0.028
Stainless Steel Cathode Mass Gain (g)	0.138	0.115	0.082
Standard Deviation SD/RSD (%)	2.62/3.83%	3.07/4.98%	3.46/5.12%

Applied voltage optimization and energy trade-offs

Applied potential provides the electrostatic driving force for ionic migration and electroreduction kinetics. Increasing voltage from 5.0 V to 10.0 V significantly elevated Ni^{2+} removal efficiency from 69.04 % to 78.94 % at pH 7.0 (t = 60.0 min) (Table 2). Raising potential further to 15.0 V provided minimal efficiency gains (plateauing at 81.20), while specific energy consumption surged over two-fold from 0.285 kWh/m³ to 0.612 kWh/m³. This inefficiency is caused by side reactions, notably parasitic cathodic hydrogen evolution ($2\text{H}_2\text{O} + 2e^- \rightarrow \text{H}_2 + 2\text{OH}^-$). Vigorous H_2 gas bubbling consumes electrical energy without contributing to metal reduction and physically dislodges electrodeposited nickel from the cathode. Thus, 10.0 V was selected as the optimal voltage balance between treatment efficiency and power utilization.

Table 2. Impact of applied voltage on electrodeposition performance and specific energy consumption (pH = 7.0, t = 60.0 min, n = 3)

Applied Voltage (U, V)	Operating Current (I, A)	Residual C_t (mg/L)	Removal Efficiency (%)	Energy Consumption (E, kWh/m ³)
5	0.175	175.54 ± 5.12	69.04	0.146
10	0.57	119.41 ± 3.10	78.94	0.285
15	1.22	106.59 ± 2.85	81.2	0.612

Electrolysis kinetics and duration

The temporal progression of the electrochemical treatment was monitored over a duration of 120.0 min under optimal conditions (pH 7.0, U = 10.0 V). During the initial 30.0 min of electrolysis, rapid nickel deposition occurred, achieving a removal efficiency of 52.15% and reducing the residual Ni^{2+} concentration to 271.30

mg/L. Extending the reaction time to 60.0 min brought the system to its peak performance, reaching a removal efficiency of 78.94% with a residual concentration of 119.41 mg/L (Fig. 2).

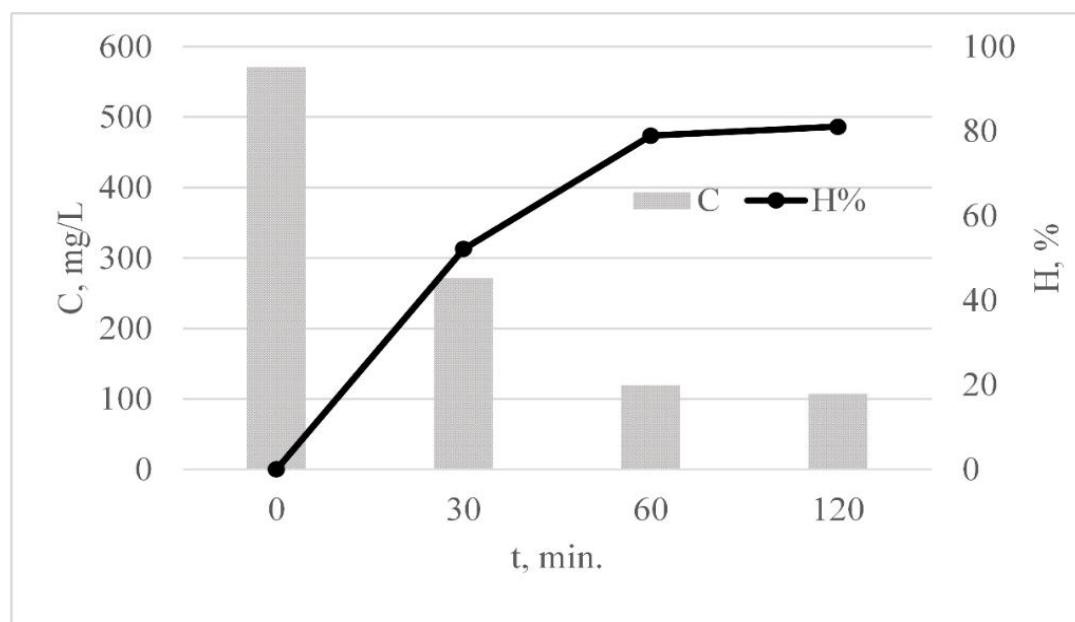


Figure 2. Investigation of electrolysis duration from 30 to 120 minutes at pH = 7.0 and U = 10 V.

The temporal removal profile was evaluated up to 120.0 min under optimized settings (pH = 7.0, U = 10.0 V). During the initial 30 min, high bulk Ni^{2+} concentration maintained rapid mass transport to the cathode, yielding 52.15 % removal. By 60 min, removal efficiency reached 78.94 % ($C_t = 119.41$ mg/L). Extending duration to 120 min yielded only an additional 2.11 % removal (81.05 %) (Table 3). As bulk concentration depletes, concentration polarization dominates, shifting reaction kinetics from activation control to mass-transfer limitation. Hence, 60.0 min represents the optimal hydraulic retention time.

Table 3. Kinetic dynamics of nickel electrodeposition over time (pH = 7.0, U = 10.0 V, n = 3)

Electrolysis Duration (t, min)	Residual Ct (mg/L)	Removal Efficiency (%)	Cumulative Energy (E,kWh/m ³)
0	567.00 ± 0.00	0	0
30	271.30 ± 4.25	52.15	0.143
60	119.41 ± 3.10	78.94	0.285
120	107.44 ± 3.05	81.05	0.57

Nickel mass balance, resource recovery, and electrode durability

A detailed mass balance was constructed under optimal operational conditions ($\text{pH} = 7.0$, U = 10.0 V, t = 60.0 min). Out of 0.895 g of total Ni^{2+} removed from the 2.0 L treated volume, gravimetric measurements showed a net cathode mass increase of 0.816 g, corresponding to a direct cathodic metal recovery efficiency (R_{cat}) of 91.2 %. The remaining 8.8 % (0.079 g) accounted for minor hydroxide precipitates settled in the reactor basin (Table 4). Graphite anode mass loss remained minimal (0.027 g per run), demonstrating high electrochemical durability and low operational degradation compared to consumable sacrificial anodes.

Table 4. Summary of optimal operating conditions, techno-economic indicators, and mass balance metrics

Operating Parameter / Performance Indicator	Selected Value / Result
Initial pH	7
Applied Voltage (U)	10.0 V

Electrolysis Duration (t)	60.0 min
Ni ²⁺ Removal Efficiency	78.94%
Residual Ni ²⁺ Concentration	119.41 mg/L
Specific Energy Consumption (E)	0.285 kWh/m ³
Cathodic Metal Recovery Rate (R _{cat})	91.2% of removed Ni ²⁺
Primary Chemical Sludge Generation	Negligible (8.8% secondary precipitate)

Practical implications, scale-up challenges, and downstream integration

While non-sacrificial direct electrolysis minimizes hazardous sludge generation and enables direct metal recovery, two key engineering limitations must be addressed. First, regarding residual concentration thresholds, single stage batch treatment yields a residual nickel concentration of 119.41 mg/L, which remains significantly above stringent regulatory discharge limits such as Vietnam's QCVN 40:2025/BTNMT Column A (0.2 mg/L), making direct discharge unfeasible without further processing. Second, concerning reactor flow hydraulics and scalability, small-scale static batch reactors (2.0 L) encounter severe mass transport constraints as bulk target ion concentrations decline. Scaling up to industrial applications requires shifting toward continuous-flow configurations, such as expanded bed or flow through 3D mesh reactors, to enhance mass transfer coefficients. To overcome these challenges, an integrated process scheme is proposed where direct electrolysis functions as a primary recovery unit to capture over 90% of high purity nickel directly at the cathode. The treated effluent is subsequently directed into downstream polishing operations, such as ion-exchange columns or nanofiltration membranes, creating a hybrid system that protects and extends the operational lifespan of the polishing media while ensuring full regulatory compliance and promoting circular resource recovery.

CONCLUSION

This study demonstrated the efficiency of an electrochemical process using a non-sacrificial graphite anode and a grade 304 stainless steel cathode for treating Ni²⁺ rich electroplating wastewater. Optimal operational parameters were identified as pH 7.0, an applied voltage of 10.0 V, and an electrolysis time of 60.0 min. Under these conditions, the system achieved a 78.94 % nickel removal efficiency, lowering residual concentrations to 119.41 mg/L with a low specific energy consumption of 0.285 kWh/m³. Mass balance evaluations confirmed that 91.2 % of removed nickel was successfully recovered directly as metallic plating on the cathode. Although the system eliminates primary toxic sludge generation, the remaining nickel concentration requires integration with downstream polishing technologies (such as ion exchange or membrane filtration) for continuous-flow industrial applications. Future research should focus on multi-factor optimization designs, long-term electrode durability studies, and scale-up trials in continuous-flow configurations.

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