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Zinc solvent extraction from waste electronic card leaching solutions

Extracción de zinc por solventes a partir de soluciones de lixiviación de tarjetas electrónicas de desecho

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Abstract

In this study, the purification of a leach solution obtained from waste electronic cards was investigated to separate zinc from base metals (Cu, Fe, and Ni) and to produce a purified $ZnSO_4$ solution through solvent extraction. A comparative evaluation was performed using Cyanex 272 and D2EHPA as extractants at 15% v/v in the organic phase, achieving zinc extraction efficiencies of 94% and 90%, respectively, at pH 3.0 and an O/A phase ratio of 1.2:1. McCabe–Thiele diagrams indicated that two theoretical extraction stages are required for Cyanex 272, whereas three stages were needed for D2EHPA. During the stripping stage, sulfuric acid (H_2SO_4) solutions at concentrations of 180 and 200 g/L were tested under varying phase ratios. Under optimal conditions, a stripping efficiency of 95% was obtained for the organic phase loaded with Cyanex 272 (O/A = 1:1), while 94% was achieved for D2EHPA under the same phase ratio.

Keywords: Zinc, Solvent Extraction, Cyanex 272, D2EHPA, Secondary Resources.

Resumen

En esta investigación, se estudió la purificación de una solución de lixiviación de tarjetas electrónicas de desecho para separar zinc de los metales base (Cu, Fe y Ni) y obtener una solución purificada de $ZnSO_4$, mediante el proceso de extracción por solventes, realizando un estudio comparativo de los extractantes Cyanex 272 y D2EHPA con una concentración al 15 % v/v en la fase orgánica, obteniendo extracciones del 94% y 90% respectivamente, a un pH 3.0 y una relación de fases ORG/AQ 1.2:1. Los diagramas de McCabe-Thiele muestran que son necesarias 2 etapas de extracción teóricas para Cyanex 272 y 3 etapas teóricas para D2EHPA. En la etapa de despojamiento se utilizó una solución de ácido sulfúrico (H_2SO_4) a una concentración de 180g/L y 200 g/L, variando las relaciones de fases, obteniendo un despojamiento arriba del 95 % para la fase orgánica con Cyanex 272, con una relación de fases de 1:1 y del 94% para la solución con D2EHPA, con una relación de fases de 1:1.

Palabras clave: Zinc, Extracción por Solventes, Cyanex 272, D2EHPA, Recursos Secundarios.

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1 Introduction

Green or circular mining has experienced notable growth in its current application, focusing on the recovery and recirculation of metals of high industrial interest. The main objective is to recover strategic metals that, due to their high demand and exploitation, are currently considered critical or difficult to access. This has opened a wide range of possibilities for the recovery of these metals through secondary raw materials, such as waste electrical and electronic equipment (WEEE) (Schaeffer *et al.* 2018). This waste has been rapidly increasing worldwide (Cayumil *et al.* 2016), becoming one of the most critical pollution problems due to the fast evolution of electronic devices, which causes electronic appliances to become obsolete for users and to end up in landfills, causing adverse environmental effects (Kiddee et al. 2013). Therefore, their recovery, recycling, and reincorporation into the supply chain seeks to have a positive environmental impact and, at the same time, it aims at mitigating the environmental effects associated with the exploitation and extraction of key minerals for the industry, such as zinc, which is fundamental in processes such as galvanization and has multiple applications in sectors such as electronics, energy, and food (Zhang *et al.* 2014).

To optimize the extraction of zinc from secondary resources, metallurgical routes that offer more efficient and sustainable recovery methods, primarily from waste, continue to be explored; one example is the waste generated by the steel industry, where research has focused on the leaching of electric arc furnace dust (EAFD) (Borda & Torres, 2021; Zhu Xiao-lin, 2019). Another area for zinc recovery from waste is the recycling of printed circuit boards (PCBs), which are essential components in the operation of electrical and electronic equipment. Their polymetallic composition makes them highly valuable for recovering base, precious, or critical metals (Rao *et al.* 2020). However, the extraction process can be complex, which is why selective methodologies and/or pretreatments for the recovery of base and precious metals must be developed in order to achieve effective extractions (Martinez-Ballesteros *et al.* 2023; Segura-Bailón & Lapidus-Lavine, 2023). For this reason, the focus of this research is on obtaining a purified zinc electrolyte solution from a pressure acid leaching solution of printed circuit boards (PCBs), aiming at recovering base metals (Cu, Ni, Zn, and Fe) in the initial stage (Martinez-Ballesteros *et al.* 2024; Martinez-Ballesteros *et al.* 2023).

To obtain this purified zinc electrolyte solution, a solvent extraction process has been chosen. This is a widely studied technique for base metals such as copper, yielding favorable results due to its selectivity for metal ions of interest, in addition to being very economical. The solvent extraction process involves two phases: an aqueous phase and an organic phase. The aqueous phase is the leach solution, while the organic phase consists of an extractant with an affinity for the target metal and a modifier in a diluent, commonly kerosene. This process is attractive due to its high efficiency and low cost (Aguayo *et al.* 2007). The selection of the extractant depends on the chemical composition of the leach solution, the metals to be extracted, and the impurities present, which constitutes a real challenge in the development of liquid–liquid extraction processes. This is especially necessary for the treatment of complex minerals or secondary resources to be carried out more efficiently and economically (Chagnes & Cote, 2010; Omelchuk *et al.* 2018).

In this work, two acidic organophosphorus extractants were used, which have been extensively studied for zinc extraction (Sun *et al.* 2019) as separating zinc from other metals has become an important industrial resource. One of them is di-2ethylhexyl phosphoric acid (D2EHPA), which exhibits high selectivity for zinc due to its cation exchange mechanism (Xie *et al.*, 2019). Some authors propose the saponification of D2EHPA to improve zinc extraction percentages, increasing recovery by up to 25% compared to unmodified D2EHPA (Tahmasebizadeh & Javanshir, 2021). Other researchers have also widely used bis(2,4,4-trimethypentyl) phosphinic acid (Cyanex 272) for zinc extraction due to its high selectivity (Li et al. 2016; Lupi & Pilone, 2020; Sun *et al.* 2019). Ahmadipour *et al.* studied the synergistic effects of these extractants combined for zinc recovery from other metals, obtaining favorable results (Ahmadipour et al. 2011). For this reason, the solvent extraction process, combined with a meticulous adjustment of the operational parameters, including solution pH, reagent concentration, and the aqueous-to-organic phase ratio (AQ: ORG; AQ refers to the aqueous phase, and ORG corresponds to the organic phase), was used to achieve optimal selectivity during metal extraction from leach solutions (Habashi, 2003). This is crucial because these solutions often contain metal ions with similar chemical behaviors, making the precise configuration of the operational conditions essential for efficient and selective separation (Sun *et al.* 2019).

The extraction (Eq. 1) and re-extraction (Eq. 2) reactions expected for zinc using an organophosphorus extractant are as follows, where R will be $(C_8H_{17})PO_2H$ (Cyanex 272) or $C_{16}H_{35}O_4P$ (D2EHPA).

$$\mathrm{ZnSO}_{4(\mathrm{AQ})} + 2\,\mathrm{RH}_{(\mathrm{ORG})} \longleftrightarrow ZnR_{2(\mathrm{ORG})} + \mathrm{H}_2\mathrm{SO}_{4(\mathrm{AQ})} \tag{1}$$

$$ZnR_{2(\mathrm{ORG})} + \mathrm{H}_2\mathrm{SO}_{4(\mathrm{AQ})} \longleftrightarrow \mathrm{ZnSO}_{4(\mathrm{AQ})} + 2\,\mathrm{RH}_{(\mathrm{ORG})} \tag{2}$$

2 Methodology

The solution obtained from the pressure acid leaching of waste electronic circuit boards (PCBs) was subjected to analysis and a purification process for the selective recovery of metals. To achieve this, the procedure was structured in four key stages.

2.1 *Pre-treatment and initial analysis*

The solution obtained from the acid leaching of electronic circuit boards to separate base metals was analyzed using an AAS instrument to determine the concentrations of Zn, Cu, Fe, and Ni (Table 1). After obtaining these data, a pretreatment was carried out. Initially, the leaching liquor was treated with magnesium oxide until it reached a pH of 4, which precipitated the iron present in the solution (Bolin & Sundkvist, 2008). This optimizes the removal of this impurity because the extractants used in this work have a high selectivity for iron (Cognis, 2008). The initial solution, with a pH of 1.30, was gradually neutralized by the addition of powdered MgO under continuous stirring to ensure uniform dispersion. The process was maintained until the pH stabilized at 4, after which the suspension was allowed to rest to promote the precipitation of solid phases. The clarified supernatant was subsequently separated and reserved for the solvent extraction experiments, while the precipitated solid was collected and stored for further characterization. Removing iron from the solution is essential. The resulting aqueous phase was analyzed by AAS, and the metal concentrations are presented in Table 1, which shows that iron was not detected.

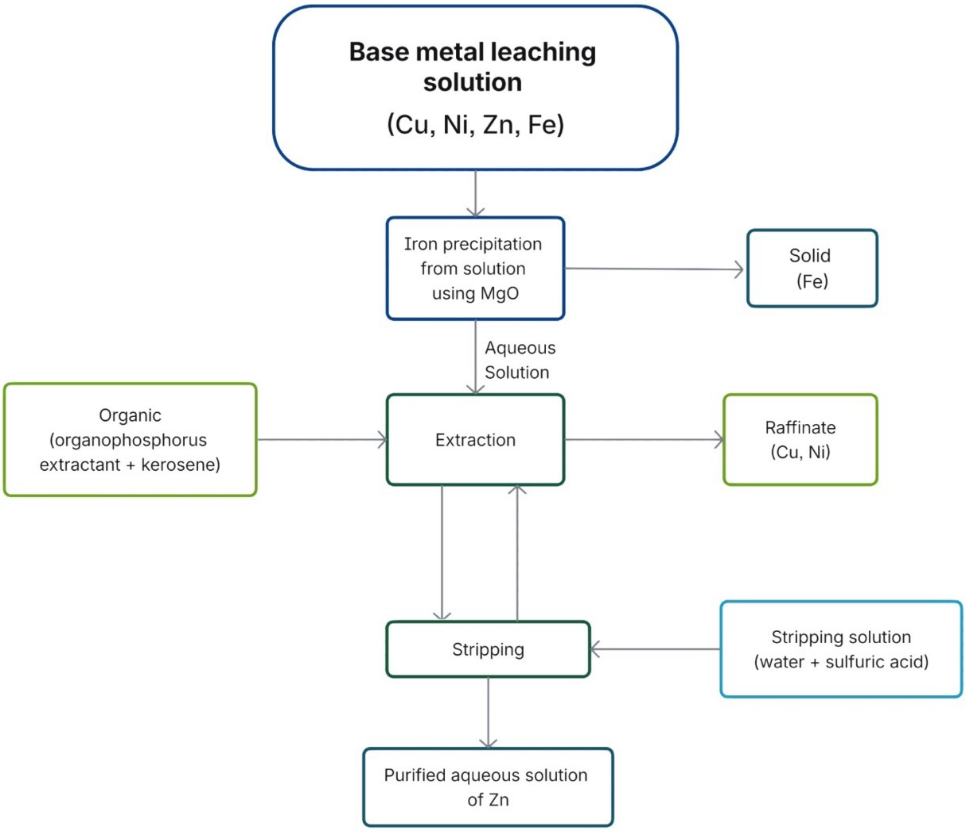

Figure 1. Experimental diagram for the solvent extraction process for a base metal leaching solution.

Table 1. Concentration of metals in solution.

Metals	Zn (mg/L)	Cu (g/L)	Ni (g/L)	Fe (g/L)
Solution before pretreatment	107.3	33.15	3.92	3.66
Solution after pretreatment	107.3	14.86	3.35	ND

Table 2. Properties characters of solvents.

Characteristic	D2EHPA	Cyanex 272	Kerosene
Molecular Formula	$C_{16}H_{35}O_4P$	$(C_8H_{17})_2PO_2H$	$C_{12}H_{26}$
Molecular Weight	322.4	290.42	170.132
Solubility in water (g/L)	<0.01	0.016	0.02
Density (g/cm^3)	0.97	0.92	0.82
Appearance	Colorless or yellowish, oily liquid	Colorless to light, amber liquid	Colorless

2.2 *Solvent extraction*

The solution obtained after iron precipitation was concentrated by applying the solvent extraction process in a 125 mL separatory funnel; the organic and aqueous phases were brought into contact and subjected to vigorous agitation to promote efficient mass transfer. The zinc concentration in the aqueous phase was analyzed to determine the optimal contact time for the process, aiming at maximizing extraction and stripping efficiency. The initial time point (t = 0) was defined as the moment when both phases were introduced into the separatory funnel, before vigorous stirring. In this funnel, the leach solution (aqueous phase) and the solvent (organic phase) were combined; the solvents used, Cyanex 272 and D2EHPA, shown in Table 2 (Ahmadipour *et al.* 2011), were added to the separatory funnel. This was followed by vigorous stirring for a fixed time of 3 minutes. Once the time had elapsed and the two phases were separated, the raffinate solution was separated from the loaded organic phase.

The experimental parameters were established as follows: the extractant concentration in the organic phase was set at 15% v/v for both extractants. The pH of the leach solution varied between 1.5, 2, 2.5, 3, and 3.5 for Cyanex 272, and between 1, 1.5, 2, 2.5, and 3 when using D2EHPA. The pH of the solution was controlled with a 1 N sodium hydroxide and 1M sulfuric acid. The AQ: ORG phase ratio varied between 0.8:1, 1:1, and 1.2:1. The concentration in the aqueous phase was determined by AAS. In contrast, the concentration in the organic phase was calculated by metallurgical balance.

The concentration of ions in the organic phase was calculated by means of a mass balance using Equation 3:

$$V_{AQ}([M]_i - [M]_f)_{AQ} = V_{ORG}([M]_f - [M]_i)_{ORG} \quad (3)$$

where V_{AQ} is the volume in the aqueous phase, V_{ORG} is the volume in the organic phase, $[M_i]_{AQ}$ represents the initial concentration of metal ions in the solution before extraction in the aqueous phase, $\left[M_f\right]_{AQ}$denotes the final concentration of metal ions after phase separation (AQ: ORG), $[M_i]_{ORG}$ represents the initial concentration of metal ions in the organic phase before extraction, and $\left[M_f\right]_{ORG}$ denotes the final concentration of metal ions after phase separation (AQ: ORG).

In Equation 3, the concentration [M] is expressed in mg/L, which is valid at any given time as well as under steady-state conditions. This mass balance was used to determine zinc concentrations in each phase during the batch experiments. Since no accumulation occurs in the system, the mass balance is also applicable under steady-state conditions. Moreover, since the system does not involve flow rates and is not continuous, this formulation is appropriate for the experimental conditions.

2.3 *Stripping*

Zinc was transferred from the loaded organic phase to the aqueous phase using sulfuric acid, with varying concentrations (180 g/L and 200 g/L). Experimentally, contact times between phases of 0.5, 1, 2, 3, 4, 5, 6, 7, and 8 minutes were tested to determine the optimal stripping time. The aqueous phase obtained was analyzed by AAS to measure the metal concentration in solution through a mass balance of the organic phase concentration (Eq. 3). The contact time for each system studied in this research was determined. The AQ: ORG phase ratio varied between 1:1, 2:1, and 5:1. This approach enabled the establishment of optimal operational parameters to maximize metal recovery during the stripping stage, ensuring the efficient transfer of metals from the organic to the aqueous phase.

2.4 *McCabe–Thiele diagram analysis*

For the generation of equilibrium isotherms, the load capacity for both organic extractants (Cyanex 272 and D2EHPA) was evaluated during the extraction and stripping stages to determine the maximum amount of zinc that can be removed from the pregnant leach solution (PLS) of PCBs in the extraction stage for each aqueous-to-organic phase ratio (AQ: ORG). The organic phase (consisting of reagent, diluent, and extracted zinc) was mixed with the PLS at various AQ: ORG phase ratios (10:1, 5:1, 2:1, 1:1, 1:2, 1:5, and 1:10) until equilibrium was reached. Each phase was then analyzed: the aqueous phase by AAS, and the organic phase concentration was calculated by mass balance (Eq. 3) (Cytec, 2010). During the stripping stage, the maximum amount of zinc that can be recovered from the organic phase was defined by mixing it with an aqueous phase containing sulfuric acid at concentrations of 180 and 200 g/L. After the AQ: ORG phase separation, the zinc concentration in each phase was analyzed. The data obtained were plotted, and McCabe–Thiele diagrams were constructed for both extraction and stripping systems. For the extraction system, zinc concentration in the organic phase was plotted on the Y-axis and zinc concentration in the aqueous phase on the X-axis. For the stripping system, zinc concentration in the organic phase was plotted on the X-axis and zinc concentration in the aqueous phase on the Y-axis.

3 Results and discussion

3.1 Extraction kinetics

Figure 2 illustrates the extraction kinetics of zinc, copper, and nickel using the extractants Cyanex 272 and D2EHPA. The data demonstrate that zinc sulfate ($ZnSO_4$) extraction efficiencies exceeded 90% for both extractants, achieving peak values of 97% with Cyanex 272 and 93% with D2EHPA at a contact time of 3 minutes. This time corresponds to the attainment of the extraction equilibrium, indicating that 3 minutes was sufficient to reach the equilibrium state under the experimental conditions.

For the extraction of $CuSO_4$ and $NiSO_4$, less than 10% extraction was achieved in both cases, with the extraction efficiency stabilizing after 2 minutes when using Cyanex 272 and after 1 minute with D2 EHPA.

3.2 Zinc extraction vs. pH

Figure 3 compares the influence of pH on the extraction behavior of zinc, copper, and nickel using Cyanex 272 (Figure 3a) and D2EHPA (Figure 3b), both at 15% v/v with an aqueous-to-organic (AQ: ORG) phase ratio of 0.8:1. With Cyanex 272, zinc extraction reached a maximum efficiency of 98% at pH 4, while copper and nickel showed limited extraction, with maximum values of 24% and 27%, respectively, both at pH 2.

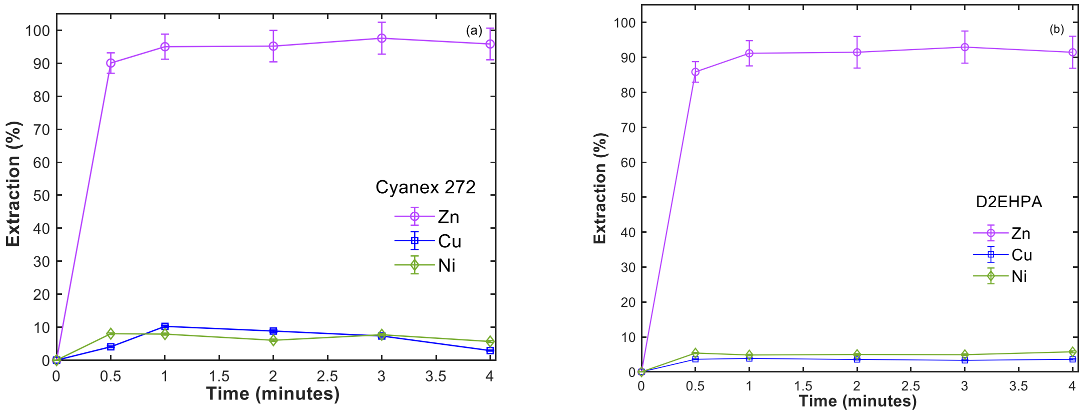

Figure 2. Extraction kinetics for zinc, copper, and nickel using (a) Cyanex 272 15% v/v AQ:ORG 1:1, pH 3; (b) D2EHPA 15% v/v AQ:ORG 1:1, pH 3.

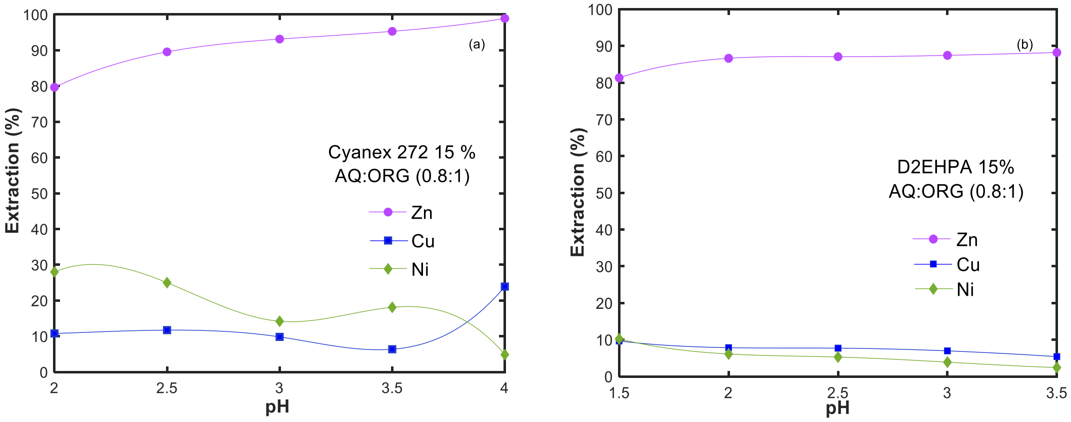

Figure 3. Zinc extraction (%) as a function of pH using (a) 15% v/v Cyanex 272 with an AQ: ORG ratio of 0.8:1 and (b) 15% v/v D2EHPA with an AQ: ORG ratio of 0.8:1.

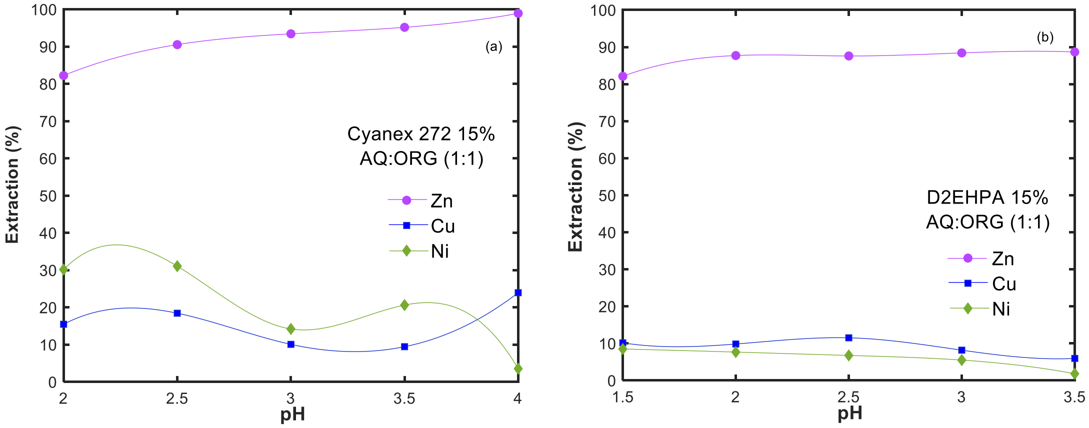

Figure 4. Zinc extraction (%) as a function of pH using (a) 15% v/v Cyanex 272 with an AQ: ORG (1:1) and (b) 15% v/v D2EHPA with AQ: ORG (1:1).

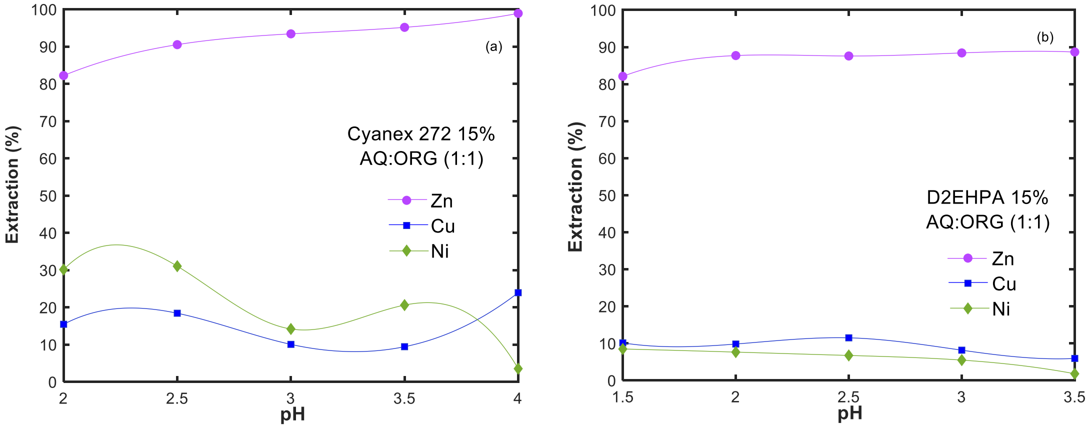

Figure 5. Zinc extraction (%) as a function of pH using (a) 15% v/v Cyanex 272 with an AQ:ORG ratio of 1.2:1 and (b) 15% v/v D2EHPA with an AQ: ORG ratio of 1.2:1.

In contrast, when D2EHPA was used under the same conditions, the maximum zinc extraction was lower, reaching 88% at pH 3.5. Copper and nickel extractions were significantly lower, with maximum values of 10% at pH 2 and 7% at pH 1.5, respectively. These results indicate that Cyanex 272 exhibits higher selectivity and efficiency for zinc than for the other base metals in the studied pH range, compared with D2EHPA.

Figure 4(a) shows the influence of pH on zinc extraction with Cyanex 272 at 15% v/v, with an AQ: ORG ratio of 1:1, achieving a maximum efficiency of 99% at pH 4. The maximum copper extraction occurred at a pH of 4, with 24% extraction, and for nickel, at a pH of 2, with 30% extraction. Figure 4(b) presents the behavior of the extractant D2EHPA 15% v/v at an AQ: ORG ratio of 1:1, where the maximum zinc extraction, 88%, was obtained at a pH of 3.5, copper extraction reached 12% at a pH of 2.5, and nickel showed its highest extraction, 8%, at a pH of 1.5.

Figure 5(a) shows the influence of pH on zinc extraction using 15% v/v Cyanex 272 at an aqueous-to-organic phase ratio (AQ: ORG) of 1.2:1, achieving a maximum efficiency of 99% at pH 4. The maximum copper extraction, 24%, occurred at a pH of 4, and nickel extraction reached 30% at a pH of 2.5. Figure 5(b) presents the behavior of 15%v/v D2EHPA at an AQ: ORG ratio of 1.2:1, where the maximum zinc extraction was 90% at a pH of 3, copper extraction reached 10% at pH 2.5%, and nickel extraction 7% at pH 2.5.

During the experimental work, it was observed that, above pH 3.5, a third interfacial phase formed for both extractants, compromising the stability of the extraction system and thus limiting its practical applicability. The optimal conditions were found at a pH of 3 for both extractants, obtaining a zinc extraction of 94 % with Cyanex 272 at AQ: ORG 1.2:1 (Fig. 5a) and 90% with D2EHPA at the same phase ratio (Fig. 5b).

According to the literature, the extractants Cyanex 272 and D2EHPA exhibit a strong affinity for divalent cations in acidic media. In agreement with these reports, it was observed that when Cyanex 272 was applied at pH values above 3, third-phase formation tended to occur, as shown in Figures 3a, 4a, and 5a. This phenomenon can be attributed to the generation of hydroxide species such as $Ni(OH)^+$, $Ni(OH)_2$, $Cu(OH)^+$, and $Cu(OH)_2$, which destabilize the organic phase. Additionally, protonation of the solution within this pH range appears to favor the occurrence of third phases, further limiting the applicability of Cyanex 272 under such conditions.

3.3 *Extraction isotherms and McCabe-Thiele diagrams*

Based on the optimal conditions obtained from the batch experimentation, isotherms and McCabe–Thiele diagrams were developed to evaluate the theoretical extraction stages. The AQ: ORG ratios were varied (1:0.8, 1:1, and 1:1.2), and the data derived guided the scaling of the experiments to a continuous system once the required extraction stages had been determined. Experimentally, the optimal conditions were found at pH 3 and an AQ: ORG phase ratio of 1.2:1 for both extractants. Figure 6(a) shows the McCabe–Thiele diagram for Cyanex 272, where theoretically two stages were required for zinc extraction. Figure 6(b) presents the McCabe–Thiele diagram for D2EHPA, indicating that three theoretical stages were needed for zinc extraction using this reagent. The theoretical stages indicated by the McCabe–Thiele diagrams, when the phase ratio was varied (AQ: ORG) to 0.8:1 or 1:1, showed no significant difference.

3.4 *Selectivity of Cyanex 272 and D2EHPA extractants*

For the calculation of the distribution coefficient and selectivity of metals (Zn, Cu, and Ni) in the aqueous solution using the extractants Cyanex 272 and D2EHPA, both at a concentration of 15% v/v, the distribution coefficient is defined as a function that depends on parameters such as temperature, pH, and the composition of the organic phase. The calculation was based on Equation (4):

$$D_M = \frac{[M]_{ORG}}{[M]_{AQ}} \tag{4}$$

where $[M]_{ORG}$ is the concentration of the metal in the organic phase and $[M]_{AQ}$ the concentration of the metal in the aqueous phase.

The selectivity factor for zinc was calculated following Equation 5:

$$S_M^{Zn} = \frac{D_{Zn}}{D_M} \tag{5}$$

where D_{Zn} is the distribution coefficient corresponding to zinc and D_M is the distribution coefficient corresponding to the other metals (copper and nickel) that were compared to zinc. From these calculations, Table 3 summarizes the distribution coefficients of the extractant Cyanex 272.

The extractant exhibited the following selectivity order:

Zn>Cu>Ni

Table 4 summarizes the distribution coefficients of the extractant D2EHPA.

The extractant exhibited the following selectivity order:

Zn>Ni>Cu.

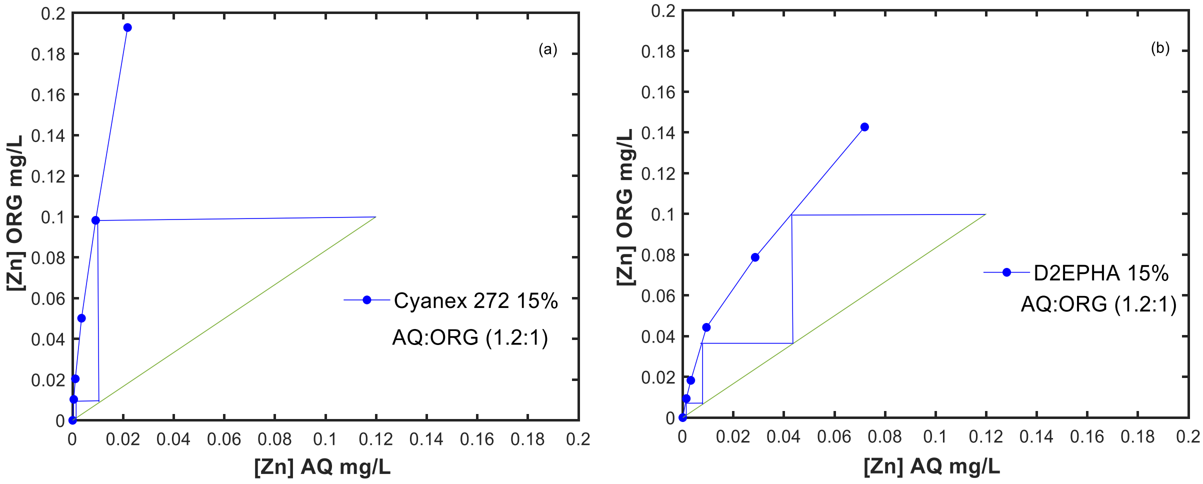

Figure 6. McCabe–Thiele diagrams using (a) Cyanex 272 at 15% v/v, AQ:ORG (1.2:1), and (b) D2EHPA at 15% v/v, AQ:ORG (1.2:1).

Table 3. Distribution coefficients and selectivity for Cyanex 272.

Metal	AQ:ORG	Extractant	$[M]_{AQ}$ (mg/L)	$[M]_{ORG}$ (mg/L)	D_M	S_M^{Zn}
Zn	1.2:1	Cyanex 272	6.6	100.6	15.2	1
Cu	1.2:1		13562	1357.7	0.10	200
Ni	1.2:1		2928	661.6	0.23	67

Table 4. Distribution coefficients and selectivity for D2EHPA

Metal	AQ:ORG	Extractant	$[M]_{AQ}$ (mg/L)	$[M]_{ORG}$ (mg/L)	D_M	S_M^{Zn}
Zn	1.2:1	D2EHPA	11.6	95.7	8.4	1
Cu	1.2:1		14016	904.3	0.06	125
Ni	1.2:1		3467	122.7	0.04	241

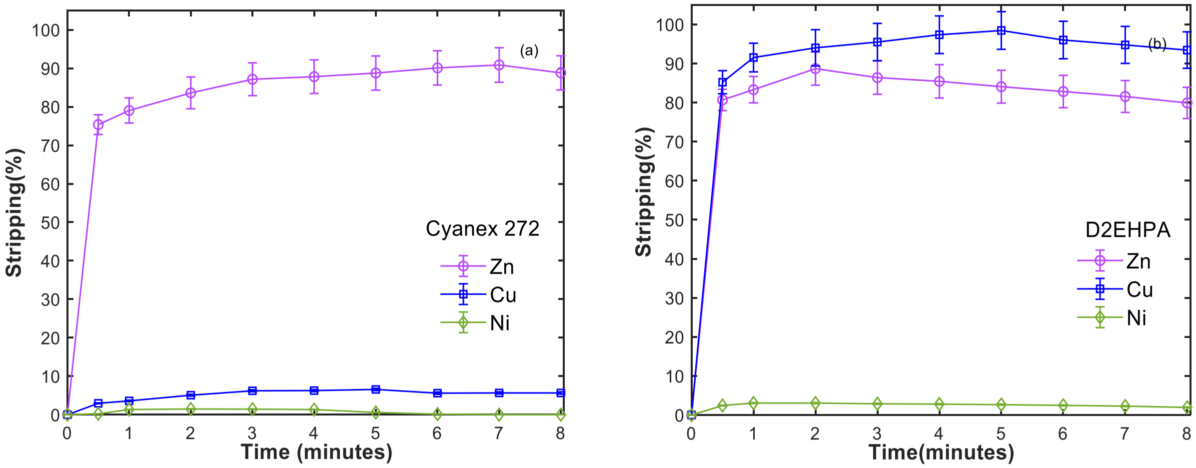

Figure 7. Stripping kinetics for zinc, copper, and nickel using H_2SO_4 at 180g/L, AQ/ORG phase ratio 1:1, extractant 15% v/v: (a) [Zn] in Cyanex 272; (b) [Zn] in D2EHPA.

3.5 Stripping kinetics

For the stripping tests, a sulfuric acid concentration of 180 g/L was used, and the contact time between phases during re-extraction was varied (0.5, 1, 2, 3, 4, 5, 6, 7, and 8 minutes). As shown in Figure 7(a), the stripping efficiency reached a maximum at 7 minutes, achieving 91% recovery of zinc contained in the solution with Cyanex 272, followed by a maximum copper recovery of 6.5% and nickel at a lower percentage of 3.8%. The latter becomes undetectable in the aqueous solution after 5 minutes. In Figure 7(b), a maximum zinc re-extraction of 88% was observed at 2 minutes. For copper, the highest re-extraction occurs after 5 minutes, reaching 98%. In contrast, nickel shows a lower percentage of 3% at 1 minute, remaining stable over time.

3.6 McCabe–Thiele analysis for stripping

McCabe–Thiele diagrams were constructed to determine the number of theoretical stages required for zinc stripping from the organic phase, examining two sulfuric acid concentrations in the aqueous phase, namely, 180 and 200g/L, and varying aqueous-to-organic phase ratios (AQ: ORG), namely, 1:1, 2:1 and 5:1. Figure 8(a) shows the steps required for stripping, when the aqueous solution has a sulfuric acid concentration of 180g/L and is put in contact with the organic phase at a 1:1 phase ratio with Cyanex 272 15%. This suggests that three theoretical stages were necessary to achieve complete zinc stripping. Figure 8(b) shows the theoretical stages required for stripping when the phase ratio was AQ: ORG of 1:1, with a concentration in the aqueous phase of sulfuric acid of 180 g/L, and the organic phase with D2EHPA 15%, where, under these established conditions, only one theoretical stage was required in the zinc stripping process.

Figure 9(a) shows the theoretical stages required for zinc stripping from the organic phase containing Cyanex 272, when in contact with an aqueous phase of H_2SO_4 at 200g/L concentration, indicating that three theoretical stages are necessary for zinc re-extraction. Figure 9(b) illustrates that for zinc stripping from the organic phase with D2EHPA under the same aqueous sulfuric acid concentration (200 g/L), only one theoretical stage is required for re-extraction. It is observed that increasing the sulfuric

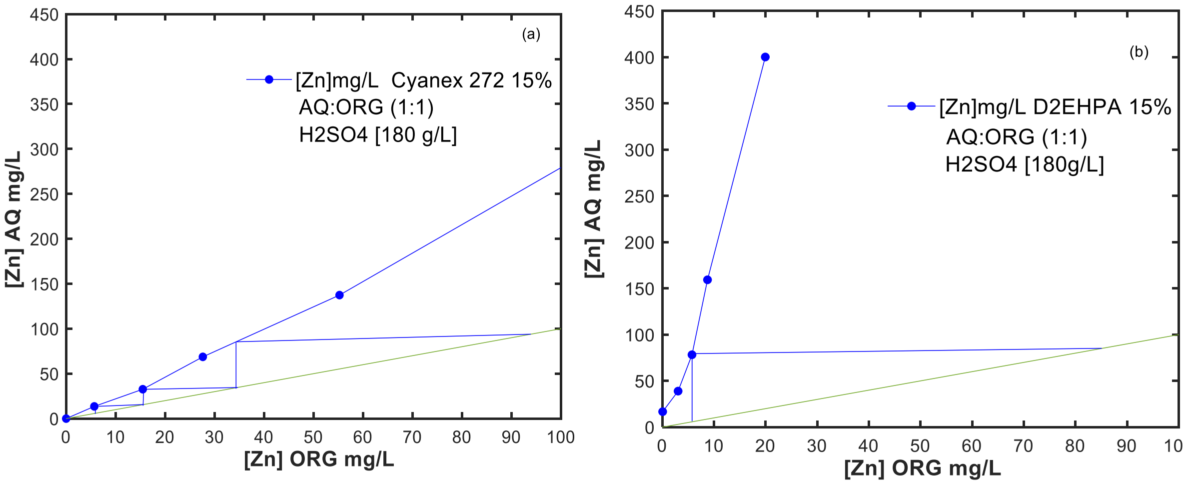

Figure 8. McCabe–Thiele diagrams for Zn stripping, AQ: ORG phase ratio (1:1), [H_2SO_4] 180g/L, 15% v/v extractant: (a) Cyanex 272; (b) D2EHPA.

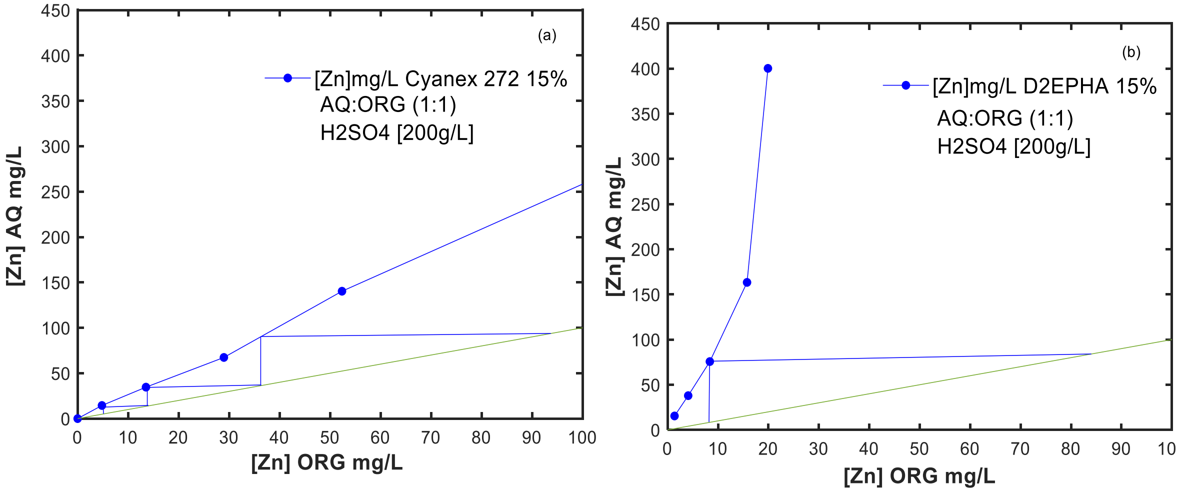

Figure 9. McCabe–Thiele diagrams for Zn stripping, AQ: ORG phase ratio (1:1), [H_2SO_4] 200g/L, 15% v/v extractant: (a) Cyanex 272; (b) D2EHPA.

acid concentration from 180 to 200 g/L does not affect the number of theoretical stages needed for zinc stripping. The optimal conditions were found at an aqueous-to-organic phase ratio of 1:1 for both Cyanex 272 and D2EHPA, as this ratio exhibited better phase separation compared to 2:1 and 5:1 for both extractants.

Conclusions

The use of organophosphate extractants, such as Cyanex 272 and D2EHPA, proved effective for the selective extraction of zinc ions from sulfuric acid solutions. Cyanex 272 exhibited higher zinc extraction efficiencies; however, its performance at elevated pH values was limited by the formation of colloidal solutions and third phases, which resulted in organic phase entrainment into the aqueous phase. This instability highlights a practical drawback of Cyanex 272 under operational conditions above pH 3. Under optimized conditions at pH 3, zinc extraction efficiency reached 94%, while copper extraction was 10% and nickel extraction was 15%, revealing the challenge of achieving complete selectivity. In comparison, D2EHPA demonstrated more stable behavior across the tested pH range, reaching a maximum zinc extraction of 90%. Copper and nickel extractions remained around 10% or less, confirming a higher selectivity for zinc under acidic conditions, despite the slightly lower overall extraction efficiency.

Once the optimal parameters were defined, namely, 3 minutes of contact time, an AQ: ORG phase ratio of 1.2:1, and a pH of 3, Cyanex 272 achieved 94% zinc extraction, with copper extraction at 8% and nickel extraction at 18%. In the subsequent stripping step, performed with 180 g/L H_2SO_4 for 7 minutes, zinc recovery from the organic phase reached 91%, while that of copper decreased to 6%

and nickel was not detectable by AAS, confirming the high efficiency of the re-extraction stage. Under similar extraction conditions, D2EHPA extracted 90% zinc, 6% copper, and 4% nickel. During stripping with 180 g/L H_2SO_4 and a contact time of 2 minutes, the zinc recovery reached 88%, underscoring the faster kinetics of D2EHPA despite its slightly lower overall efficiency.

McCabe–Thiele diagrams provided additional insight into stage requirements. At optimal conditions of pH 3 and an AQ: ORG phase ratio of 1.2:1, zinc extraction with Cyanex 272 required two theoretical stages, whereas D2EHPA required three stages. In the stripping step, increasing the sulfuric acid concentration from 180 to 200 g/L did not significantly influence the number of stages, indicating that 180 g/L was sufficient for efficient operation. Under these conditions, stripping with Cyanex 272 required three theoretical stages, while D2EHPA required only one stage to achieve zinc recoveries above 90%. These findings suggest that Cyanex 272 offers higher extraction efficiency, but with the drawback of co-extraction and potential third-phase formation. In contrast, D2EHPA provides greater operational stability and faster stripping performance, albeit with a slightly lower zinc loading capacity.

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