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Liquid–liquid extraction of phosphorus from sulfuric acid solution using benzyl dimethyl amine

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Abstract: This study addresses the liquid–liquid extraction behavior of phosphorus from a sulfuric acid solution using benzyl dimethyl amine (BDMA) in kerosene. The extraction equilibria investigated with varied BDMA concentrations could reveal the formation of $3[\text{BDMA}] \cdot [\text{H}_3\text{PO}_4]$ complex in the organic phase. The thermodynamic properties determined at various temperatures indicated that the process was exothermic with a calculated enthalpy (ΔH^\ominus) of $-24.0 \text{ kJ} \cdot \text{mol}^{-1}$. The organic-to-aqueous phase (O/A) volume ratio was varied to elucidate the quantitative extraction of phosphorus. The McCabe–Thiele diagram plotted for the extraction isotherm was validated for the requirement of three counter-current stages in the extraction at an O/A volume ratio of 2.0/3.5. The back-extraction of phosphorus from the loaded organic phase was quantitatively achieved by contacting $4.0 \text{ mol} \cdot \text{L}^{-1} \text{ H}_2\text{SO}_4$ solution in three stages of counter-current contact at an O/A volume ratio of 3/2. This study can be applied to remove phosphorus from the sulfuric acid leach solutions of monazite processing, and many other solutions.

Keywords: solvent extraction; phosphorus; benzyl dimethyl amine (BDMA); sulfuric acid solution; McCabe–Thiele diagram

1. Introduction

The presence of phosphorus in many industrial solutions and effluents has long been viewed as a significant problem in terms of high-purity end products and sustainable processing. Particularly, phosphorus remains in a solution by the sulfuric acid processing of phosphorus-bearing minerals of rare earth elements (REEs) including monazite ((Ce,La,Y)PO₄) and xenotime (YPO₄), and other rock phosphates including fluorapatite are of significant concern [1–4]. Because of the adversity caused by phosphorus in the H₂SO₄ leach solution either using acid baking followed by water leaching or roasting followed by acid leaching [5–6], nowadays, alkali roasting is preferably applied to extract REEs from phosphorous-bearing minerals. However, alkali processing consumes high amount of energy and large volumes of water and acid to neutralize the free alkalinity. Besides the extraction of REEs, phosphorus-bearing domestic and industrial wastewater discharged into the environment is responsible for algae bloom and eutrophication in water bodies [7–8]. Generally, a small amount of phosphorus can

cause a substantial algae growth. Hence, phosphorus removal becomes more crucial than nitrogen removal to prevent algae bloom and eutrophication [9].

Numerous chemical, microbial, and bio-chemical methods were employed to separate and remove phosphorus from various solutions [10]. However, they could only be specifically applied to a particular type of solution. For example, precipitation with iron, calcium, or aluminum salts is largely applied for sludge treatment; sedimentation or filtration is applied to wastewater treatment [11]; crystallization is applied to obtain hydroxyapatite (Ca₅(OH)(PO₄)₃) [12]. Moreover, microorganisms that require phosphorus for their metabolic growth [13–14] have also been used. However, none of these methods can be employed to separate and remove phosphorus from leach solutions that have significantly higher phosphorus contents than wastewater or effluents. Liquid–liquid extraction (LLE) using a solvo-chemical technique is a potential process that can handle a wide range and nature of solutions because of the inherent characteristics of LLE. Notably, LLE uses a certain quantity of a chosen organic solvent (with an appropriate diluent) to extract the desired solute spe-

cies from the aqueous solution via complexation in the organic phase until an equilibrium is reached [15–16]. LLE is widely accepted in hydrometallurgy involving the separation, enrichment, and recovery of REEs from phosphorus minerals. However, very limited reports exist on the LLE of phosphorus, particularly from the sulfuric acid solution, albeit its co-existence in sulfuric acid processing is well-known [5–6]. In a recent study, Mu *et al.* [17] used Cyanex 923 for knowing the extraction behavior in the presence of phosphorus; however, phosphorus was found to be co-extracted with cerium. Therefore, the separate handling of phosphorus becomes imperative.

The LLE of phosphorus from sulfuric acid solution by using benzyl dimethyl amine (BDMA) in kerosene has been investigated in this study. Although a few studies used BDMA to remove phosphorus from wastewater [9,18], they were not systematic and did not deal with solutions with high phosphorus content (dealt with only 30 mg·L⁻¹). However, a 380 mg·L⁻¹ phosphorus-bearing solution considered as an aqueous feed in this study; the solution may come from the sulfuric acid leach solution processing of monazite. Accordingly, parameters including BDMA concentration, solution acidity of H₂SO₄, temperature, and organic-to-aqueous phase (O/A) volume ratio were varied; subsequently, stripping was conducted for regenerating the organic phase and performing phosphorus recovery back into the aqueous phase.

The novelty of this study can be defined in terms of the possible recovery of high-purity phosphorus instead of the removal techniques it uses. The regeneration ability of the organic solvent used in this study helps reduce the process cost by its re-use in the extraction-stripping process. The process is environmental friendly because it saves a significant amount of water, which is required in usual alkali cake-washing; additionally, the process also saves the energy required in the slow filtration of the sticky cake. Notably, the P-containing alkali cake is usually handled as an impurity and not for the recovery purpose as the potential of this study. Therefore, the present route of LLE can be considered a sustainable solution to deal with phosphorus impurity in various aqueous streams because of the associated benefits of environmental benignness, resource utilization, low-cost energy-saving operation, and recyclability of reagents. The LLE technique can be employed in the extractive metallurgy of rock-phosphate minerals including monazite, where high-purity phosphates can also be efficiently recovered as the by-product of the process, thereby leading to a sustainable process.

2. Experimental

2.1. Materials

A stock solution containing 380 mg·L⁻¹ of phosphorus was prepared by dissolving trisodium phosphate salt

(Na₃PO₄, purity 96%, supplied by Sigma-Aldrich, Germany) in a 2.0-mol·L⁻¹ sulfuric acid solution (using H₂SO₄, 95% purity, supplied by Daejung, Korea). An organic solvent of BDMA (purity ≥99%, density of 0.9 g·mL⁻¹ at 25°C, supplied by Sigma-Aldrich, Germany) was prepared in distilled kerosene (supplied by Junsei Chemical Co., China). The samples were diluted in 0.5 mol·L⁻¹ hydrochloric acid solution (using HCl, purity 36.46%, supplied by Daejung, Korea) for the analysis of phosphorus in aqueous solutions. All the aqueous solutions were prepared in distilled water, and the reagents were used without further purification.

2.2. Methods

Unless specified, all the experiments were performed in a 100-mL glass separatory funnel by equilibrating an equal volume, i.e., 30 mL, of the aqueous and organic phases, each for 10-min duration under the room temperature of (20 ± 1)°C. Both the phases were settled for the next 10 min after the equilibration time, and the aqueous solution (raffinate) was withdrawn to analyze the phosphorus content using inductively coupled plasma spectroscopy (ICP-OES, iCAP 7400 Duo, Thermo Scientific, USA). The concentration of the phosphorus extracted in the organic phase was determined using the mass balance Eq. (1). Subsequently, the extraction efficiency (*E*) was calculated using Eq. (2), while the distribution ratio (*D*) was determined using Eq. (3), as follows:

$$C_P(\text{organic}) = C_P(\text{initial}) - C_P(\text{raffinate}) \quad (1)$$

$$E = \frac{C_P(\text{organic})}{C_P(\text{initial})} \times 100\% \quad (2)$$

$$D = \frac{C_P(\text{organic})}{C_P(\text{raffinate})} \quad (3)$$

where *C_P*(organic) is the concentration of phosphorous extracted into organic phase, *C_P*(initial) represents the concentration of phosphorous in leach liquor, and *C_P*(raffinate) is the concentration of phosphorous remaining in aqueous phase after extraction.

The stripping study was conducted using a phosphorous-loaded organic generated via counter-current extraction (CCE). The loaded organic was washed with water at an O/A volume ratio of 2/1 before contacting with sulfuric acid solutions of various concentrations. The equilibration, settling, and sampling were performed similar to the extraction studies, while the stripping efficiency (*S*) was calculated as follows:

$$S = \frac{C_P(\text{strip})}{C_P(\text{organic})} \times 100\% \quad (4)$$

where *C_P*(strip) represents the concentration of phosphorous that was stripped from organic phase.

The characterization of the organic phase was performed using Fourier-transform infrared spectroscopy (NICOLET

380 FT-IR, Thermo Electron Corp., USA) using the wavenumber that ranged from 400 to 4000 cm^{-1} .

3. Results and discussion

3.1. Solution chemistry of phosphorus

The solution chemistry of phosphorus is fairly complicated and dominated by P^{5+} . Under highly acidic conditions, phosphorus is involved with three acidic protons and thus exists as $\text{H}_3\text{PO}_4(\text{aq})$ [19], which changes with decreasing acid concentrations to $\text{H}_2\text{PO}_4^-(\text{aq})$, $\text{HPO}_4^{2-}(\text{aq})$, and $\text{PO}_4^{3-}(\text{aq})$ (see Fig. 1). Besides these predominant species, some polymeric compounds including pyrophosphate ions, i.e., $\text{P}_2\text{O}_7^{4-}(\text{aq})$, and triphosphate ions, i.e., $\text{P}_3\text{O}_{10}^{5-}(\text{aq})$, exist in the meta-stable form. In low-valance states under anaerobic conditions, phosphorus also exists as $\text{PO}_3^{3-}(\text{aq})$ and $\text{PO}_2^{3-}(\text{aq})$; however, these states are not considered in our study because of the dominance of P^{5+} in the solution in terms of their thermodynamic properties, i.e., enthalpy of formation, $\Delta_f H^\ominus$: $\text{H}_3\text{PO}_4(\text{aq})$, $-1288.3 \text{ kJ}\cdot\text{mol}^{-1}$; $\text{H}_2\text{PO}_4^-(\text{aq})$, $-1296.3 \text{ kJ}\cdot\text{mol}^{-1}$;

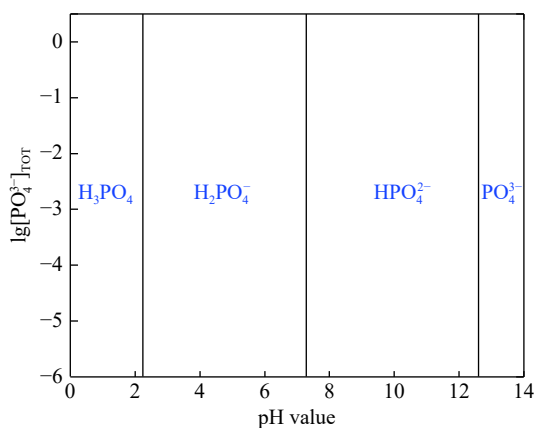


Fig. 1. Speciation diagram of phosphorus as a function of pH value for the phosphorus concentration of $0.01 \text{ mol}\cdot\text{L}^{-1}$, temperature of 298 K, and pressure of 101.325 kPa.

$\text{HPO}_4^{2-}(\text{aq})$, $-1292.1 \text{ kJ}\cdot\text{mol}^{-1}$; and $\text{PO}_4^{3-}(\text{aq})$, $-1277.4 \text{ kJ}\cdot\text{mol}^{-1}$ [19–20].

3.2. Effect of extractant concentration

The extraction behavior of phosphorus with an organic extractant was investigated using different BDMA concentrations in the range of $0.05\text{--}0.50 \text{ mol}\cdot\text{L}^{-1}$ at an O/A volume ratio of 1. The results in Fig. 2 revealed that phosphorus extraction could be improved by increasing the BDMA concentration in the organic phase and simultaneous decrease of the phosphorus content in the raffinate. This can be described by the shifting distribution curve with respect to readily available extraction sites for phosphorus complexation [21–22]. The availability of readily available sites with higher extractant population potentially increases the complexation between the solute and solvent [16]. The extraction efficiency of phosphorus increased from 6% to approximately 90% upon increasing the extractant concentration from 0.05 to $0.4 \text{ mol}\cdot\text{L}^{-1}$ and finally reached approximately 94% with $0.5 \text{ mol}\cdot\text{L}^{-1}$ of BDMA. The inhibited progress in phosphorus extraction for the BDMA concentration above $0.4 \text{ mol}\cdot\text{L}^{-1}$ can be ascribed to the increased protonation of the organic extractant [16].

The complexation of phosphorus with BDMA was determined by plotting a $\lg D$ versus $\lg[\text{BDMA}]$ graph. The plot yielded a straight line with a regression coefficient (R^2) of 0.98, as shown in Fig. 2. Furthermore, the slope of the straight line indicated the involvement of three BDMA molecules to extract one mole of phosphorus in the organic phase. Looking at the phosphorus speciation under acidic conditions (see Fig. 1), phosphorus remained as H_3PO_4 under the studied condition. Therefore, H_3PO_4 may be extracted via the following two-step reaction [23]: (i) intermediate salt formation of the amine extractant, and (ii) loading of the H_3PO_4 species by displacing the H_2SO_4 from the organic salt. The extraction reactions can be expressed as follows:

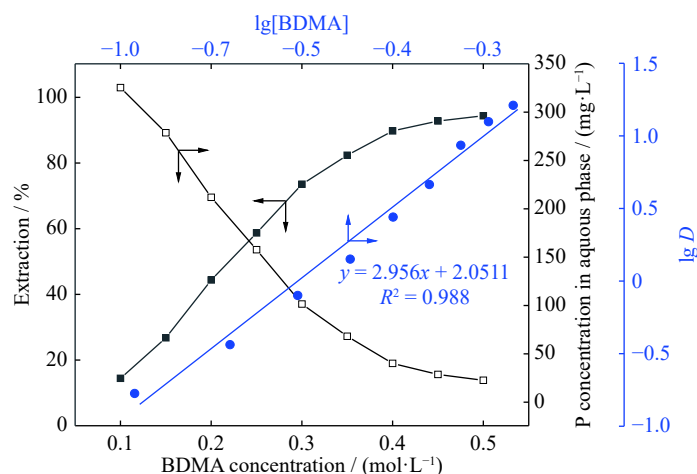
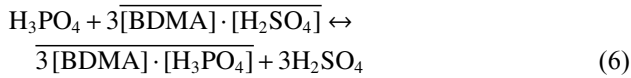


Fig. 2. Phosphorus extraction behavior as a function of the extractant concentration, and the plot of $\lg D$ versus $\lg[\text{BDMA}]$.



Notably, the water molecule participated in bridging for the extraction of phosphorous from a low acidic solution in the organic phase; while, the extracted compound formed a complex with the amine extractant at a high acid concentration [24]. The Fourier transform infrared spectroscopic (FTIR) analysis (see Fig. 3) supported such a complexation, where a band assigned to PO_4^{3-} at 1048 cm^{-1} , along with the vibrational bands at 1466 and 1531 cm^{-1} , was observed. A small shift of the C–N band and stretching from 1096 to 1084 cm^{-1} [25] indicated the direct involvement of the C–N group into the complexation with PO_4^{3-} and the presence of –OH group in the complex.

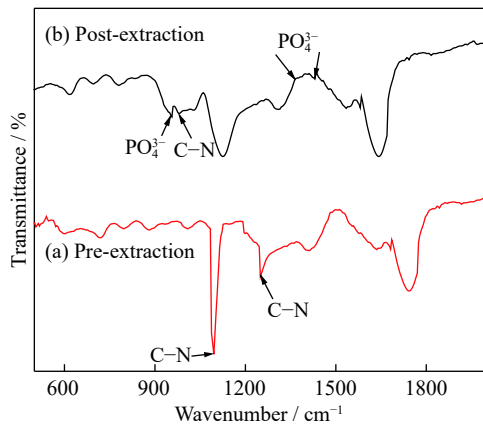


Fig. 3. FTIR spectra of the fresh organic containing BDMA and phosphorus-loaded organic phase.

3.3. Effect of temperature

The effect of temperature on phosphorus extraction was investigated by varying the temperature from 20 to 50°C while using $0.5 \text{ mol}\cdot\text{L}^{-1}$ BDMA and an O/A volume ratio of 1. Because of the possible degradation of amine solvents at high temperatures, this study was kept limited for temperatures up to 50°C . The results plotted in Fig. 4 show that an increasing temperature adversely affects the extraction efficiency. The extraction efficiency decreased from 94% to 86.5% upon increasing the temperature from 20 to 50°C . This extraction efficiency behavior reveals that the ability of metal-binding sites of BDMA is destroyed with increasing temperature. Furthermore, the thermodynamic properties of the extraction equilibrium were determined using the van't Hoff equation in terms of the phosphorus distribution (D) in organic and aqueous phases, as follows [15,22]:

$$\lg D = -\frac{\Delta H^\ominus}{2.303RT} + \frac{\Delta S^\ominus}{2.303R} \quad (7)$$

The slope and intercept values of $\lg D$ versus $1/T$ were used to calculate the apparent changes in both enthalpy and entropy (ΔH^\ominus and ΔS^\ominus , respectively), which were obtained to be $-24.0 \text{ kJ}\cdot\text{mol}^{-1}$ and $-59.1 \text{ J}\cdot\text{K}^{-1}\cdot\text{mol}^{-1}$, respectively. These values were substituted in Eq. (8) to calculate the change in the standard Gibbs free energy as follows:

$$\Delta G^\ominus = \Delta H^\ominus - T\Delta S^\ominus \quad (8)$$

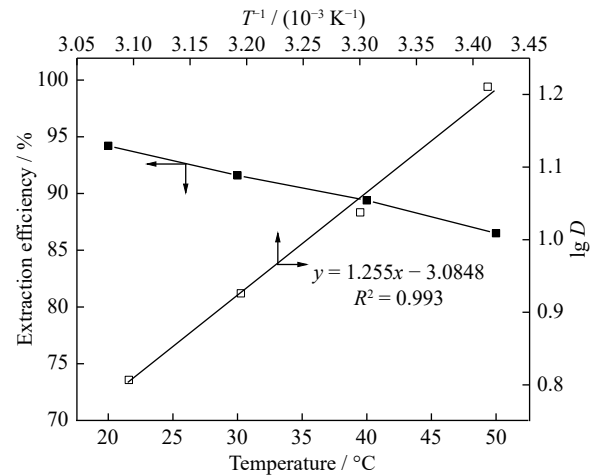


Fig. 4. Phosphorus extraction behavior as a function of temperature using $0.5 \text{ mol}\cdot\text{L}^{-1}$ BDMA in the organic phase, and the van't Hoff plot for the distribution of phosphorus versus $1/T$.

The values of ΔG^\ominus calculated at various temperatures are presented in Table 1. The analyses of the thermodynamic properties revealed that the phosphorus extraction with BDMA was a spontaneous and exothermic process, which involved the formation of an ordered extracted species in the organic phase [15,22].

Table 1. Thermodynamic properties of the extraction process, as a function of temperature

Temperature / K	Enthalpy, $\Delta H^\ominus / (\text{kJ}\cdot\text{mol}^{-1})$	Entropy, $\Delta S^\ominus / (\text{J}\cdot\text{K}^{-1}\cdot\text{mol}^{-1})$	Gibbs free energy, $\Delta G^\ominus / (\text{kJ}\cdot\text{mol}^{-1})$
293			-6.7
303	-24.0	-59.1	-6.1
313			-5.5
323			-4.9

3.4. Effect of O/A volume ratio

Phosphorus extraction as a function of the O/A volume ratio (varied from 1/3 to 3/1) was investigated using $0.5 \text{ mol}\cdot\text{L}^{-1}$ BDMA in the organic phase. The results showed that the extraction efficiency significantly increased from 66% to more than 98% within the varied range of the volume phase ratios in a single contact. Using the phase-ratio data, an extraction isotherm was determined by plotting the McCabe–

Thiele diagram, as shown in Fig. 5. It was revealed that the maximum efficiency could be achieved in three stages of CCE at an O/A volume of 2.0/3.5.

Furthermore, a batch simulation study was conducted with three stages CCE until the steady-state was achieved. The equilibrium data agreed well with the extraction isotherm and experimental data. A typical mass balance data of the three stages of CCE after reaching the steady-state is shown in Fig. 6, which resulted in 648 mg·L⁻¹ phosphorus with loaded organic and leaving <10 mg·L⁻¹ phosphorus into the

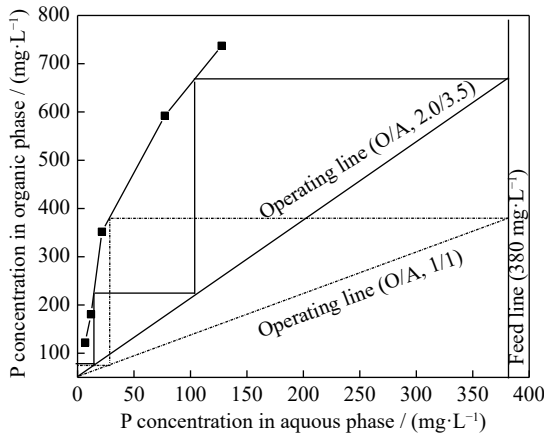


Fig. 5. McCabe–Thiele diagram for phosphorus extraction using 0.5 mol·L⁻¹ BDMA in the organic phase and 380 mg·L⁻¹ phosphorus in the aqueous feed.

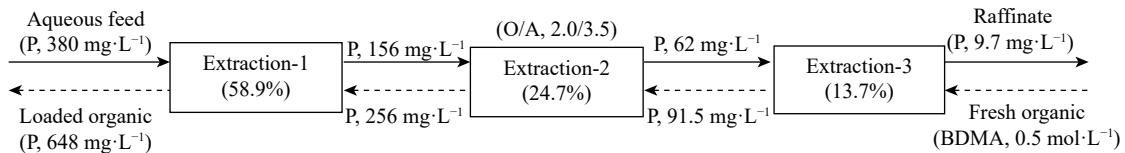


Fig. 6. Distribution of phosphorus between the organic and aqueous phases during the three stages of CCE at an O/A volume of 2.0/3.5 using 0.5 mol·L⁻¹ BDMA in the organic phase and 380 mg·L⁻¹ phosphorus in the aqueous feed.

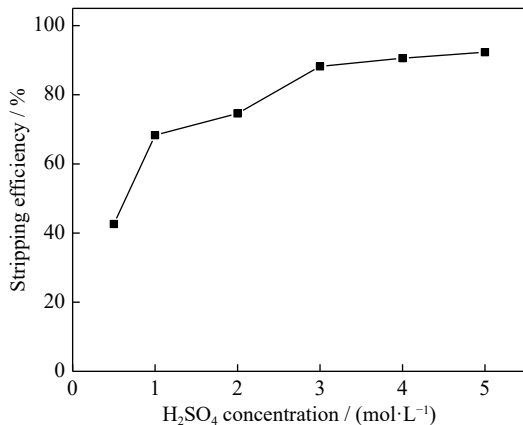


Fig. 7. Stripping behavior of phosphorus as a function of sulfuric acid concentration using a loaded organic bearing 648 mg·L⁻¹ phosphorus contacted at an O/A volume ratio of 1.

raffinate. Thus, the generated loaded organic with enriched phosphorus was subsequently used for the stripping studies in the next sets of experiments.

3.5. Phosphorus stripping from the loaded organic

Phosphorus stripping from the loaded organic was conducted at various H₂SO₄ solution concentrations in the range of 2.0–5.0 mol·L⁻¹. The results in Fig. 7 showed that approximately 90% of the phosphorus could be stripped with 4.0 mol·L⁻¹ H₂SO₄ solution, while it was only 42.6% with 0.5 mol·L⁻¹ H₂SO₄. No significant improvement was observed while contacting the loaded organic with a >4.0 mol·L⁻¹ H₂SO₄ solution.

Furthermore, to obtain the quantitative stripping of phosphorus and regeneration of the organic phase, a stripping study was performed at different O/A volume ratios, varied from 1/5 to 5/1. The data obtained were used to plot the McCabe–Thiele diagram, as shown in Fig. 8. It was revealed that the maximum stripping could be achieved in three stages of counter-current stripping while maintaining the O/A volume ratio at 3/2. A batch simulation of three-stage counter-current stripping was performed until the steady-state was achieved after the seventh cycle. The equilibrium data agreed well with the stripping isotherm and experimental values with approximately 99% yield of 961 mg·L⁻¹ phosphorus in stripped solution, thereby revealing the potential of this study.

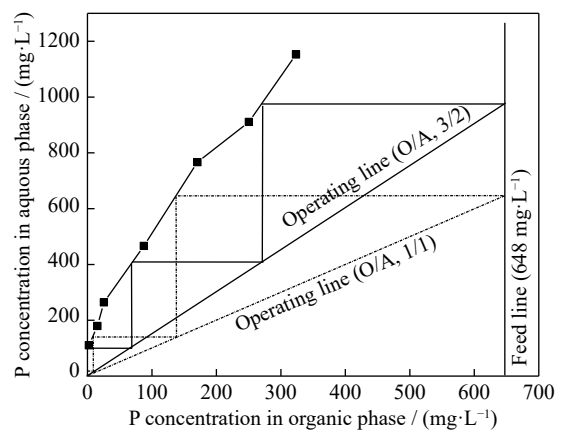


Fig. 8. McCabe–Thiele diagram for stripping for a loaded organic containing 648 mg·L⁻¹ phosphorus in 0.5 mol·L⁻¹ BDMA while contacting with a 4.0 mol·L⁻¹ H₂SO₄ solution.

4. Conclusion

The solvo-chemical extraction of phosphorus from sulfuric acid solution was successfully demonstrated using BDMA as the potential extractant. The parametric variation study showed that the extraction efficiency increased upon increasing the BDMA concentration in the organic phase; however, the extraction efficiency decreased with increasing temperature. The slope analysis of the phosphorus distribution versus organic extractant concentration, along with the solution chemistry of phosphorus, revealed that the species extracted in the organic phase was $3[\text{BDMA}] \cdot [\text{H}_3\text{PO}_4]$. The thermodynamic properties determined by this study indicated that the extraction process was exothermic and spontaneous. The extraction and stripping isotherm obtained by plotting the McCabe–Thiele diagram was also validated in the counter-current mode of contacts, yielding approximately 99% of phosphorus recovery in a $4.0 \text{ mol} \cdot \text{L}^{-1} \text{ H}_2\text{SO}_4$ solution. The regeneration of the organic extractant and the recovery of an enriched phosphorus solution ($961 \text{ mg} \cdot \text{L}^{-1}$) proved advantageous for the extraction process.

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