

Phosphorus removal by lime-natural mineral dissolved solutions

Joohyun Kim^{1,2}, Sunho Yoon¹, Jueun Jung¹ and Sungjun Bae^{*1}

¹Department of Civil and Environmental Engineering, Konkuk University,
120 Neungdong-ro, Gwangjin-gu, Seoul 05029, Republic of Korea

²School of Chemical and Biological Engineering, Institute of Chemical Process (ICP),
Seoul National University, 1 Gwanak-ro, Gwanak-gu, Seoul, 08826, Republic of Korea

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Abstract. In previous studies, solely ferric (Fe^{3+}) and calcium (Ca^{2+}) ions were commonly used for removal of $\text{PO}_4\text{-P}$ (considered as T-P in this study) in wastewater via chemical precipitation. Herein, the removal of total phosphorus (T-P) in wastewater was performed using various mineral and lime dissolved solutions. The dissolution kinetics of different minerals (feldspar, olivine, elvan, illite, sericite, and zeolite) and lime was compared and used their solutions for T-P removal of real wastewater. The highest T-P removal (almost 90%) was obtained by the lime dissolved solution and followed by zeolite, illite, feldspar, and others. We observed a significant co-relationship (R of 0.96) between the amount of initial Ca^{2+} and T-P removal. This was induced by formation of hydroxyapatite-like mineral via Ca-P precipitation reaction at high pH solution. Furthermore, additional removal of suspended solid (SS) and chemical oxygen demand (COD) was achieved by only lime dissolved solution. Finally, the lime-feldspar dissolved solutions were prepared at different ratios (10-50%), which showed a successive T-P removal up to two times by samples of 40 and 50%.

Keywords: Ca dissolved solution; feldspar; lime; mineral; phosphorus

1. Introduction

Due to the rapid growth of population in the world, the fertilizer demand for food production has been increased dramatically every year (Burakova and Bakšienė 2021, Vaccari *et al.* 2019). Because phosphorus (P) is contained in various agricultural chemicals (e.g., surfactant, fertilizer, and pesticides), the increase of P concentration in water bodies has been continued (Shin *et al.* 2023, Kwak and Yun 2020). High concentration of P in aquatic environment can cause severe problems such as eutrophication, depletion of oxygen level, and toxic effect to aquatic organisms (Li *et al.* 2016). Therefore, US Environmental Protection Agency (US EPA) and European Union (EU) recommend the total P concentration of lake under 0.05 mg/L and 0.01 mg/L for prevention of eutrophication (Loganathan *et al.* 2014).

To manage the aquatic P concentration, many efforts have put on development of efficient and cost-effective methods via biological (Desireddy *et al.* 2022), chemical (Li *et al.* 2020, Shin *et al.* 2016), and physical treatments (Nir *et al.* 2018, Choi *et al.* 2019, Hong *et al.* 2020). Among them, a chemical approach (e.g., coagulation and precipitation) has been commonly applied to the P removal due to its fast reaction and easy operation (Nir *et al.* 2018). In particular, ferric (Fe^{3+}) and calcium (Ca^{2+}) ions have been well-known as efficient precipitants. For application of ferric ion, relatively high chemical cost, need of low pH effluent, and difficulty of recycling of Fe-P precipitate are considered as main drawbacks (Thistleton *et al.* 2002). On the other hand, calcium ions revealed the rapid reaction

with cost-effective application and requirement of high pH effluent as its advantage and disadvantage, respectively (Morse *et al.* 1998).

Previous studies demonstrated the highly efficient removal of P (almost 99% removal) by calcium containing chemicals such as chloride with sodium hydroxide and $\text{Ca}(\text{OH})_2$ (Hosni *et al.* 2008, 2007). However, both chemical methods also showed some drawbacks because the used chemicals are expensive and highly alkaline. Thus, an alternative should be developed to reduce the chemical consumption and lower the unreacted chemical sludge after the reaction.

To address the problem above, this study first compared the possibility of P removal by non-commercial reagent based Ca solution with the lime dissolved solution. Various minerals (i.e., feldspar, olivine, elvan, illite, sericite, and zeolite) were used as alternative sources of ions to prepare the solutions containing Ca or other ions. The main objectives of this study were i) to find out the maximum dissolved metallic species (Al, Si, Cr, Fe..etc) and ions (i.e., cation and anion) in each mineral suspensions and lime dissolved solution, ii) evaluate the removal efficiency of P by the dissolved solutions, iii) demonstrate the positive relationship of Ca^{2+} with P removal efficiency, iv) compare the efficiency with a commercial PAC using real wastewater, and v) demonstrate the potential use of lime-mineral dissolved solution for efficient T-P removal.

2. Experimental

2.1 Chemicals and materials

Lime (CaO) and natural minerals were obtained from Gundo Co. Korea and different mining sites in South Korea,

*Corresponding author, Professor,
E-mail: bsj1003@konkuk.ac.kr

Table 1 Final concentration of elements and dissolved ions after 24-h dissolution (tap water). Experimental condition: mineral concentration = 10 g/L in 500 mL of tap water, mixing rate = 150 rpm, mixing time = 24 h, N.D = Non-detectable, Detection limit = 0.5 mg/L (ICP-OES) and 0.1 mg/L (IC)

	ICP-OES (mg/L)				IC (mg/L)						
	Al	Si	Cr, Cu, Fe, Mn, Ni, Ti, Zn	Na ⁺	K ⁺	Ca ²⁺	Mg ²⁺	Cl ⁻	NO ₃ ⁻	SO ₄ ²⁻	
feldspar	N.D	2.11	N.D	21.1	14.1	N.D	N.D	58.5	N.D	31.6	
olivine	N.D	N.D	N.D	16.2	33	N.D	N.D	52.4	N.D	33.7	
elvan	N.D	2.87	N.D	19.7	2.4	N.D	N.D	55.4	N.D	24.7	
red clay	N.D	N.D	N.D	16.9	4.9	N.D	N.D	57.8	20.8	25.6	
illite	1.01	1.07	N.D	19.7	5.4	N.D	N.D	55.4	21.0	33.3	
lime	1.1	3.03	N.D	16.9	1.5	866	N.D	47.1	N.D	20.5	
sericite	N.D	1.61	N.D	17.8	19.6	125	N.D	66.7	22.8	N.D	

respectively. The real wastewater was obtained from one of wastewater plant near Incheon in South Korea. The characteristics of raw wastewater was investigated before conducting experiments, which revealed a 435 mg/L TS, 33~67 mg/L SS, 74~108 mg/L COD, 8.2~9.9 mg/L T-P, and pH 7.70~8.02. Deionized water (DI; 18.3 MΩ) was obtained from an ultrapure filtration system (HUMAN POWER I+ Water purification system).

2.2 Dissolution experiment and batch experiment for T-P removal

A batch experiment was conducted for dissolution experiment using 1 L of beaker. An exact amount of lime and mineral (10 and 1 g/L) was transferred to the beaker and mixed with water at 150 rpm for 24 h. The mineral dissolved solutions were prepared by withdrawing the sample after 24 h reaction and filtering it through a 0.2 μm PTFE filter. A batch experiment was conducted for the T-P removal by i) lime, ii) each mineral, and iii) lime-feldspar dissolved solutions. A commercial coagulant (i.e., Poly-aluminium chloride, PAC, 17 wt% of Al₂O₃) was also used for test of T-P removal in this study. A 25 mL of 10% of diluted dissolved solutions transferred to the beaker containing 275 mL of wastewater. Then, the solution was rapidly mixed at 150 rpm for 5 min and slowly mixed at 20 rpm for 20 min.

2.3 Analytical methods

The concentrations of T-P, SS, and COD was measured by the HACH standard kits using a spectrophotometer (DR3900, HACH) and the detection limits are 0.06, 5 and 3 mg/L, respectively. The aqueous solution was measured after filtering the suspension with 0.2 μm syringe filter (Whatman). To monitor the dissolution kinetics of Al, Si, and other metallic elements, the solution was analyzed by an inductively coupled plasma optical emission spectrometry (ICP-OES, Thermo). For determination of anions in dissolved solutions, the solution sample filtered using 0.2 μm syringe filter was analyzed by a ion chromatography (IC) (Metrohm, 883 Basic IC plus) equipped with a compact autosampler (Metrohm, 863 Compact IC) and anion column (Shodex IC Anion Sep No.82504A). Mixture

of Na₂CO₃ (3.5 mM) and NaHCO₃ (3.5 mM) was prepared for IC eluent. The concentration of cations was measured using cation column (Metrosep c4-150/4.0, Metrohm AG). A mixture of nitric acid (34 mM) and dipicolinic acid (14 mM) was used as the IC eluent (Yoon *et al.* 2021).

3. Results and Discussion

3.1 Dissolution kinetics in 10 and 1 g/L of suspensions

Fig. 1 shows the variation of metal and ion dissolution during 24-h reaction in each mineral and chemical suspension (10 g/L) prepared in tap water. Although the amount of mineral and chemical used for the dissolution experiment was high, there was no significant dissolution of Al, Si and other metallic components (i.e., Cr, Cu, Fe, Mn, Ni, Ti, and Zn). On the other hand, we observed a dramatic increase in ion concentrations in early dissolution stage, then tended to reach their equilibrium concentrations. Overall, noticeable dissolution amount of cations (i.e., Na⁺, K⁺, Ca²⁺) and anions (i.e., Cl⁻, NO₃⁻, SO₄²⁻) was obtained after 24 h-reaction. Particularly, the highest Ca²⁺ (866 mg/L) was observed in the lime suspension (Table 1) and followed by sericite (126 mg/L) and zeolite (8.6 mg/L), whereas the concentration of other cations and anions were slightly different in each mineral/chemical suspension.

Before the T-P removal experiment (anion P), the amount of used mineral was reduced from 10 g/L to 1 g/L to find out the potential use of mineral/chemical-dissolved solution as cation solution for removal of T-P in real secondary treated wastewater. Table 2 shows the concentration of dissolved elements and cations after the 24-h reaction in each mineral suspension (1 g/L) prepared in DI water. Compared to the 10 g/L of samples, almost no Al, Si, and metals were detected with significant low concentration of cations. However, the concentration of Ca²⁺ (722 mg/L) in lime suspension was slightly smaller than that of 10 g/L, indicating that 1 g/L of calcite is enough to prepare the Ca²⁺ rich solution with several hundred mg/L concentration. This is because that lime reaction with water can form calcium hydroxide (CaO + H₂O → Ca(OH)₂) and the calcium hydroxide can be dissolved in water (i.e.,

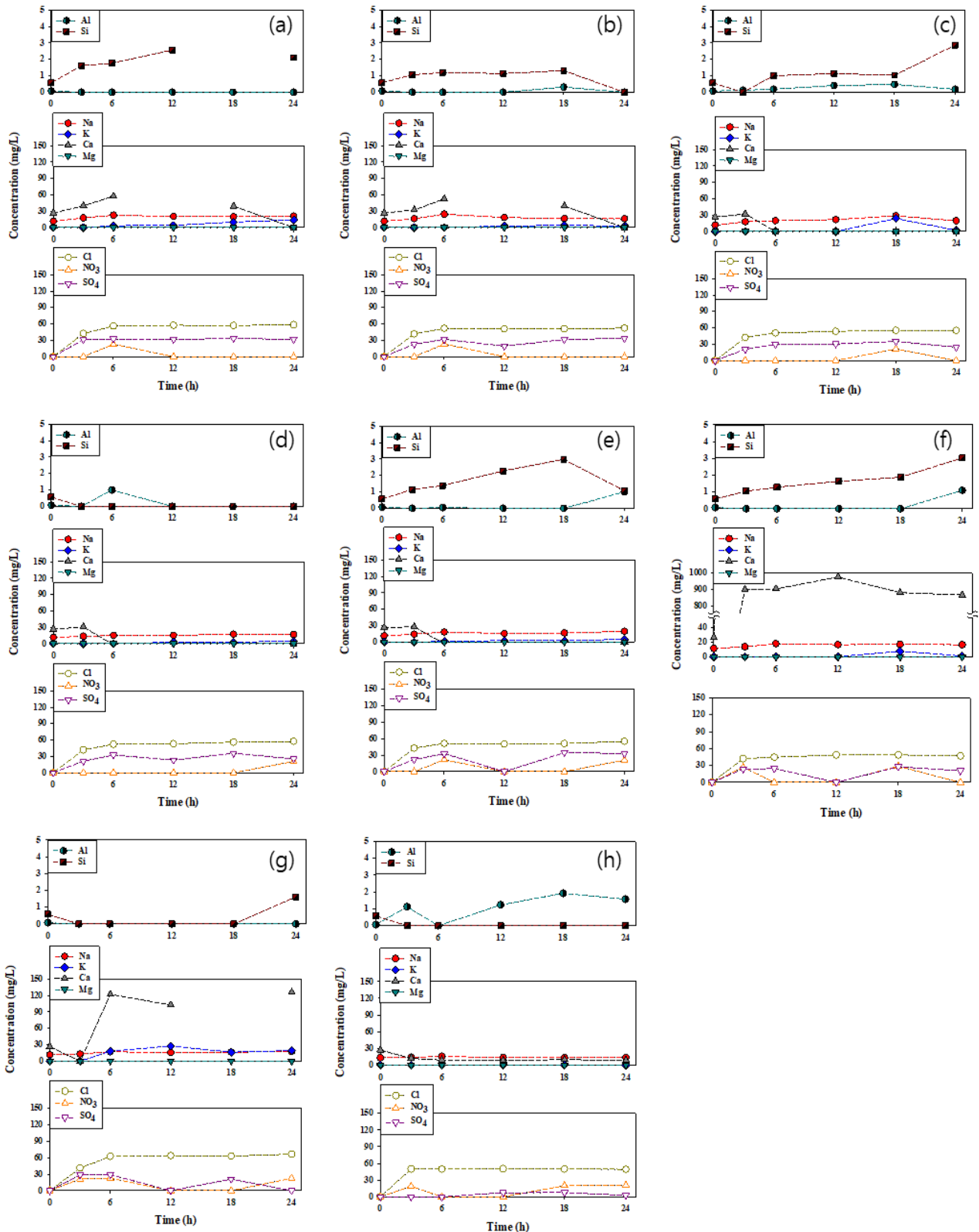


Fig. 1 Dissolution kinetics of dissolved elements and ions in different mineral suspensions; (a) feldspar, (b) olivine, (c) elvan, (d) red clay, (e) illite, (f) lime, (g) sericite, and (h) zeolite. Experimental condition: mineral concentration = 10 g/L in 500 of tap water, mixing rate = 150 rpm, mixing time = 24 h

$\text{Ca}(\text{OH})_2 \rightarrow \text{Ca}^{2+} + 2 \text{OH}^-$, solubility in water = 1.73 g/L at 20 C°, producing an alkaline solution higher than pH 12.5 (Rumble 2018).

3.2 Removal of T-P by mineral/lime dissolved solutions

Fig. 2 shows the total amount of T-P concentration

Table 2 Final concentration of elements and dissolved cations after 24-h dissolution. Experimental condition: mineral concentration = 1 g/L in 500 mL of DI water, mixing rate = 150 rpm, mixing time = 24 h, N.D = Non-detectable, Detection limit = 0.5 mg/L (ICP-OES) and 0.1 mg/L (IC)

	ICP-OES (mg/L)			IC (mg/L)			
	Al	Si	Cr, Cu, Fe, Mn, Ni, Ti, Zn	Na ⁺	K ⁺	Ca ²⁺	Mg ²⁺
feldspar	1.94	N.D	N.D	2.1	N.D	0.6	2.3
olivine	N.D	N.D	N.D	0.6	N.D	8.4	2.6
elvan	N.D	N.D	N.D	2.6	N.D	2.9	N.D
red clay	N.D	N.D	N.D	0.9	N.D	0.8	2.8
illite	N.D	N.D	N.D	N.D	N.D	14	6.3
lime	N.D	N.D	N.D	24.3	2.4	722.4	2.5
sericite	N.D	N.D	N.D	62	N.D	0.3	3.6
zeolite	N.D	N.D	N.D	7.1	N.D	20.1	3.3

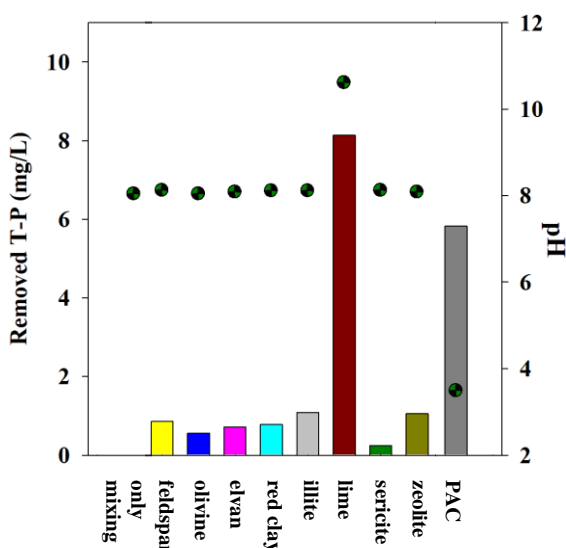


Fig. 2 T-P removal in wastewater by various mineral/lime dissolved solutions and a commercial coagulant (i.e., PAC). Experimental condition: Volume of wastewater = 275 mL, [T-P]₀ = 8.76 ~9.48 mg/L, Volume of added mineral/lime solution (10% diluted solution) = PAC = 25 mL (17 wt% of Al₂O₃), mixing at 150 rpm for 5 min and slow mixing at 20 rpm for 20 min

removed in real wastewater by various mineral/lime dissolved solutions and their final pH values after the precipitation. Almost negligible removal of T-P in control experiment (only mixing), while lesser than 1 mg/L of T-P was removed by using most of mineral dissolved solutions, except the case of lime. The lime dissolved solution revealed around 8 mg/L of T-P removal, indicating almost 90% of removal efficiency of T-P in wastewater. Interestingly, the T-P removal by PAC was much lower than that of lime dissolved solution. It should be also noted that addition of lime dissolved solution into wastewater increased the suspension pH higher than 9, which is opposite from the case of PAC showing the pH drop lesser than 4. In alkaline pH range (8~12), phosphorus could be present as HPO₄⁻ or PO₃⁴⁻ and these P species can react with the dissolved Ca²⁺ in lime dissolved solution. The high pH could improve the Ca-P precipitation reaction to form hydro-

Table 2 Final concentration of elements and dissolved cations after 24-h dissolution. Experimental condition: mineral concentration = 1 g/L in 500 mL of DI water, mixing rate = 150 rpm, mixing time = 24 h, N.D = Non-detectable, Detection limit = 0.5 mg/L (ICP-OES) and 0.1 mg/L (IC)

Lime to feldspar (wt%)	Na ⁺ (mg/L)	K ⁺ (mg/L)	Ca ²⁺ (mg/L)	Mg ²⁺ (mg/L)
0 %	12.7	2.4	36.2	35.6
10 %	15.7	7.7	310.6	36
20 %	13.6	9.9	700.5	35.4
30 %	15.3	9	1012	37.3
40 %	14.2	8.6	1159.4	36.5
50 %	12.9	8.3	1237.3	35.3

xyapatite-like mineral (Ca₅(PO₄)₃(OH) or Ca₁₀(PO₄)₆(OH)₂) (De Rooij *et al.* 1984, Nikolenko *et al.* 2020).

To find out the driving force of enhanced T-P removal by lime dissolved solution, we analyzed the co-relationship between the T-P removal amount and each ion concentration (Fig. 3). A significant co-relationship with R of 0.96 was obtained with the Ca²⁺ ions, whereas other ions did not show any co-relationship in this study. The result indicate that the Ca²⁺ with high pH were the key factor for T-P removal in this study.

3.3 Additional removal of SS and COD by mineral/lime dissolved solutions

Fig. 4 shows the additional removal of SS and COD by various mineral/lime dissolved solutions. For SS removal (Fig. 4a), all the mineral dissolved suspensions showed 50~60% of removal efficiency which was similar to that of control experiment (just mixing). This indicates that formation of SS floc may occur by the physical force. In contrast, lime dissolved solution showed almost 96% of SS removal, indicating the enhanced flocculation of SS by Ca²⁺ ions. For COD removal, we observed the highest removal efficiency in lime dissolved solution and followed by zeolite, sericite, olivine, and others. The enhanced COD removal by lime dissolved solution may be caused by the

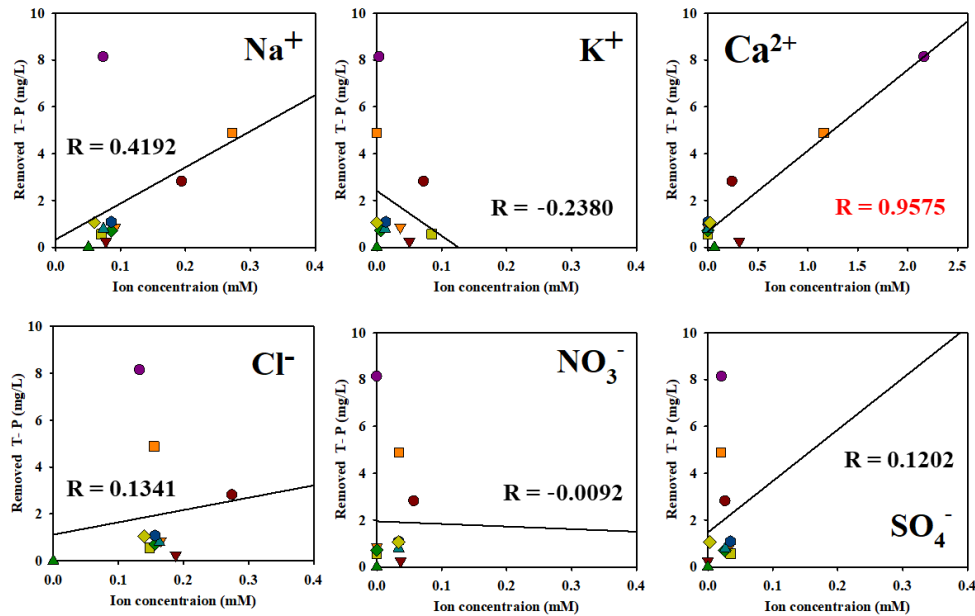


Fig. 3 Co-relationship between the T-P removal amount and each ion concentration

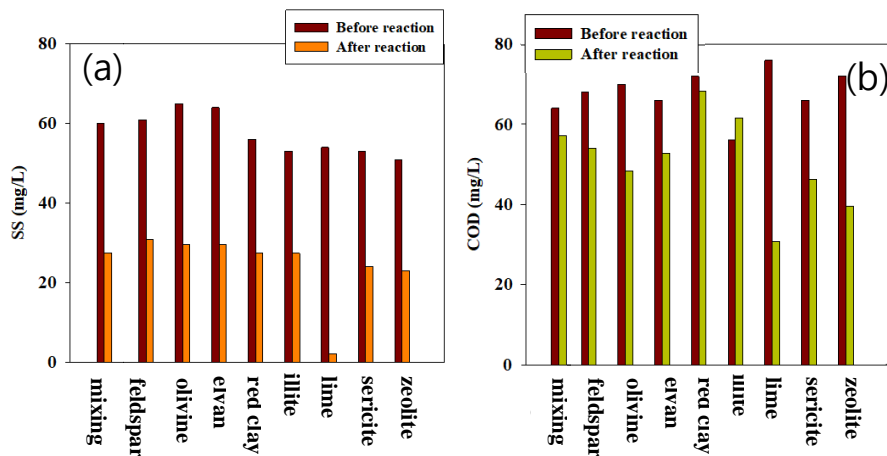


Fig. 4 Removal of (a) SS and (b) COD by various mineral/lime dissolved solutions. Experimental condition: Volume of wastewater = 275 mL, Volume of added mineral/lime solution (10% diluted solution) = 25 mL, mixing at 150 rpm for 5 min and slow mixing at 20 rpm for 20 min

enhanced SS removal via coagulation and precipitation processes (Al-Shannag *et al.* 2012). The results show that lime dissolved solution can effectively remove not only the T-P but also the SS and COD in real wastewater.

3.4 Successive removal of T-P by lime-feldspar dissolved solution

To find out the optimal usage of lime for preparation of Ca²⁺ dissolved solution and reduce the wt% of lime consumption, we prepared the lime-feldspar dissolved solutions in different wt% ratio. Feldspar was selected because it is the most cost-effective mineral with satisfying the dissolution of other cations to maintain the ionic strength of solution after the Ca loss. As expected from the results above, the Ca²⁺ concentration continuously increased to 1237 mg/L as the lime portion increased to half concentration in total (Table 3). The prepared solutions

were used for the T-P removal in wastewater (Fig. 5(a)). We observed the increase in T-P removal and the suspension pH as the lime ratio increased and reached to the maximum value (~8.5 mg/L) in this study. Figure 5(b) shows that the amount of precipitate also increased at higher ratio of lime to feldspar.

Fig. 6 shows the successive T-P removal in wastewater by different lime-feldspar dissolved solutions. The result indicates that 20 and 30% of lime contents were not enough to proceed the secondary treatment, while a successful secondary treatment was observed by 40 and 50% of experiments. Limited Ca²⁺ content may influence the T-P removal in 20 and 30%, which could be overcome by higher ratio of lime content in this study. However, there is no clear co-relationship between the total Ca²⁺ content and removal amount of T-P in this experiment, indicating the presence of other influencing factors such as pH variation-P speciation, involvement of other cations.

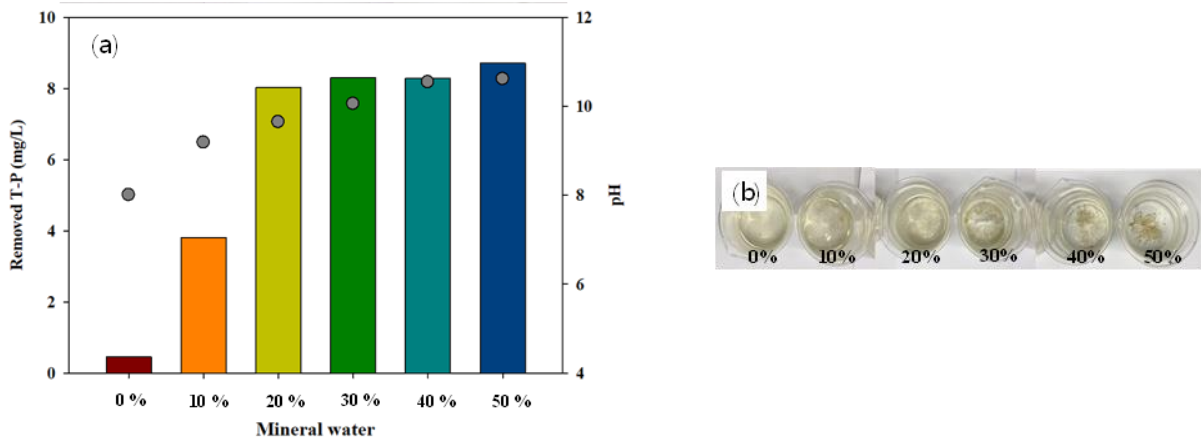


Fig. 5 T-P removal in wastewater by lime-feldspar dissolved solutions prepared in different ratio. Experimental condition: Volume of wastewater = 450 mL, $[T-P]_0 = 8.76 \sim 9.48$ mg/L, Volume of added solution (10% diluted solution) = 50 mL, mixing at 150 rpm for 5 min and slow mixing at 20 rpm for 20 min

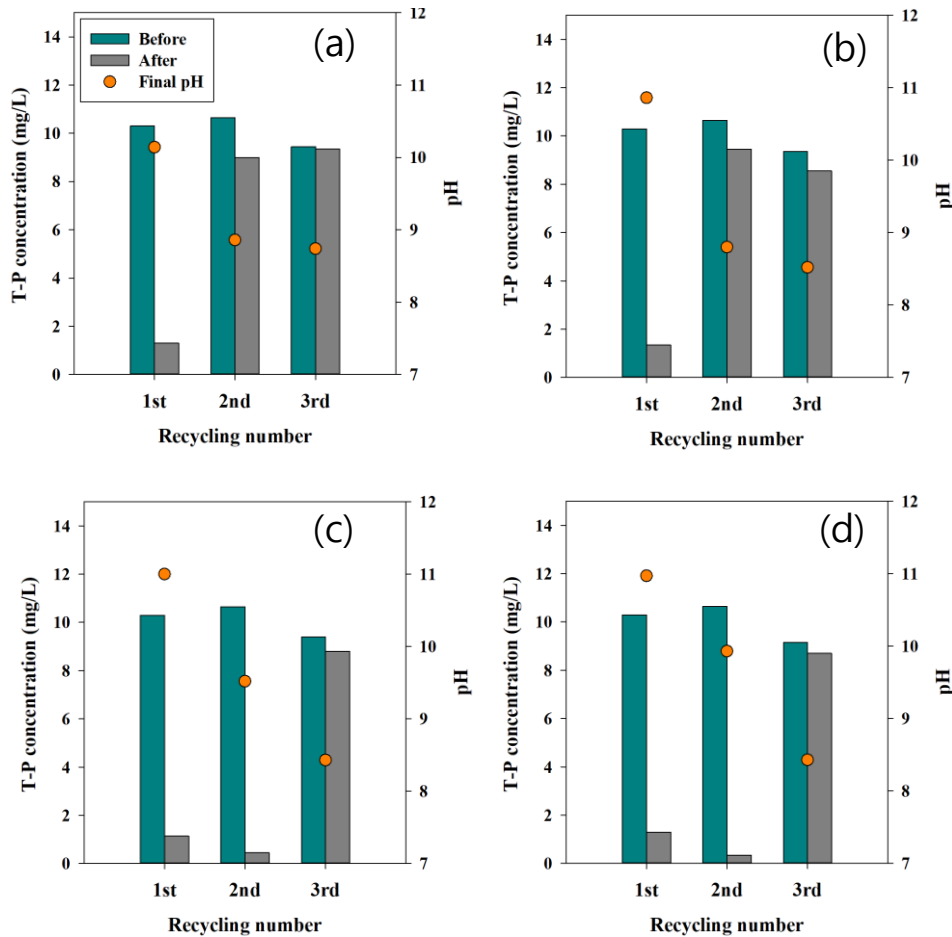


Fig. 6 Successive T-P removal in wastewater by lime-feldspar dissolved solutions prepared in different ratio ((a) 20, (b) 30, (c) 40, and (d) 50%). Experimental condition: Volume of wastewater = 450 mL, $[T-P]_0 = \sim 10$ mg/L, Volume of added solution (10% diluted solution) = 50 mL, mixing at 150 rpm for 5 min and slow mixing at 20 rpm for 20 min

4. Conclusions

The potential use of lime/mineral dissolved solution was examined for effective removal of T-P in wastewater. Compared to insignificant removal efficiency in mineral

dissolved solutions, lime dissolved solution showed almost 90% of initial T-P removal. High pH and dissolved Ca^{2+} can form the hydroxyapatite-like mineral $(Ca_5(PO_4)_3(OH))$ or $(Ca_{10}(PO_4)_6(OH)_2)$, which can also simultaneously remove the SS and COD in wastewater. The lime-feldspar dissolved

solution can be reused twice because of the limited Ca content in prepared sample, but further study should be done to find out other influencing parameters governing the efficiency of T-P removal in real wastewater. Since the alkalinity plays an important role in T-P removal, limited success can be achieved depending on the ambient pH of the wastewater for practical applications. The results obtained from this study can highlight a new insight for use of lime/mineral dissolved solution for management of T-P in wastewater instead of direct use of commercial chemical reagents which can produce lots of unreacted chemical sludge.

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