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A new viewpoint of lime/mineral dissolved solution for removal of phosphorus and the corresponding mechanism in wastewater

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Abstract. The possibility of using lime/mineral solvent solutions has been investigated to effectively remove T-P from wastewater. The lime solvent solution showed an initial T-P removal efficiency of about 90% compared to the less efficient mineral solvent solution removal. High pH and dissolved Ca2+ can form hydroxyapatite minerals (Ca5(PO4)3(OH) or Ca10(PO4)6(OH)2 and can also remove SS and COD from wastewater. Feldspar dissolution solution can be reused twice because the Ca limited sample content provided, but further research is needed to discover other influencing parameters that control the T-P removal efficiency in real wastewater. Because it plays an important role of alkalinity in T-P removal, the success rate is limited. In practical applications, it is obtained according to the pH value wastewater in the environment. The results obtained in this study can highlight new insights on the use of limestone/dissolved mineral solutions to control T-P in wastewater, instead of directly using commercial chemical agents that can produce large amounts of unreacted chemical sludge.

Keywords: artificial intelligence tool; assessment; deep learning; sustainability; water resource

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 Bakšiene 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 $Ca(OH)_2$ (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.

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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_+	Ca2+	Mg2+	Cl^-	NO3-	SO42-
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

2. Materials and methods

2.1 Chemicals and materials

Lime (CaO) and natural minerals were obtained from Gundo Co. Korea and different mining sites in South Korea, 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 Al_2O_3) 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).

2.4 Learning auxiliary

For each incoming input pattern $\underset{x}{\rightarrow}$, the spatial firing strength F_j (t) is used as the criterion for determining whether a new rule should be generated. Initially, there are no rules in the intrinsic network. For the first incoming datum $\underset{x}{\rightarrow}$ (0), a new rule is generated with an initial center and a width of a Gaussian membership function as follows:

$$m^1 = x_i(0), \sigma^1 = \sigma_{\text{initial}}$$

where $\sigma_{initial}$ is a predefined value that determines the width of each fuzzy set. For a subsequent incoming datum $\underset{x}{\rightarrow}(t)$, we calculate the maximum spatial firing strength $\underset{x}{\rightarrow}$ (t) as the indicator of whether a new rule should be generated

$$\mathrm{I} = \arg max F^j(t) \,, 1 \le \mathrm{j} \le \mathrm{R(t)}$$

$$m_i^{R(t+1)} = \mathrm{xi(t)}, \sigma_i^{R(t+1)} = \beta \cdot |xi(t) \cdot m_i^I|$$

The parameter-learning phase proceeds after the structure learning phase for each datum.

$$E(t+1) = \frac{1}{2}\Big(y(t+1) - y^d(t+1)\Big)^2$$

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

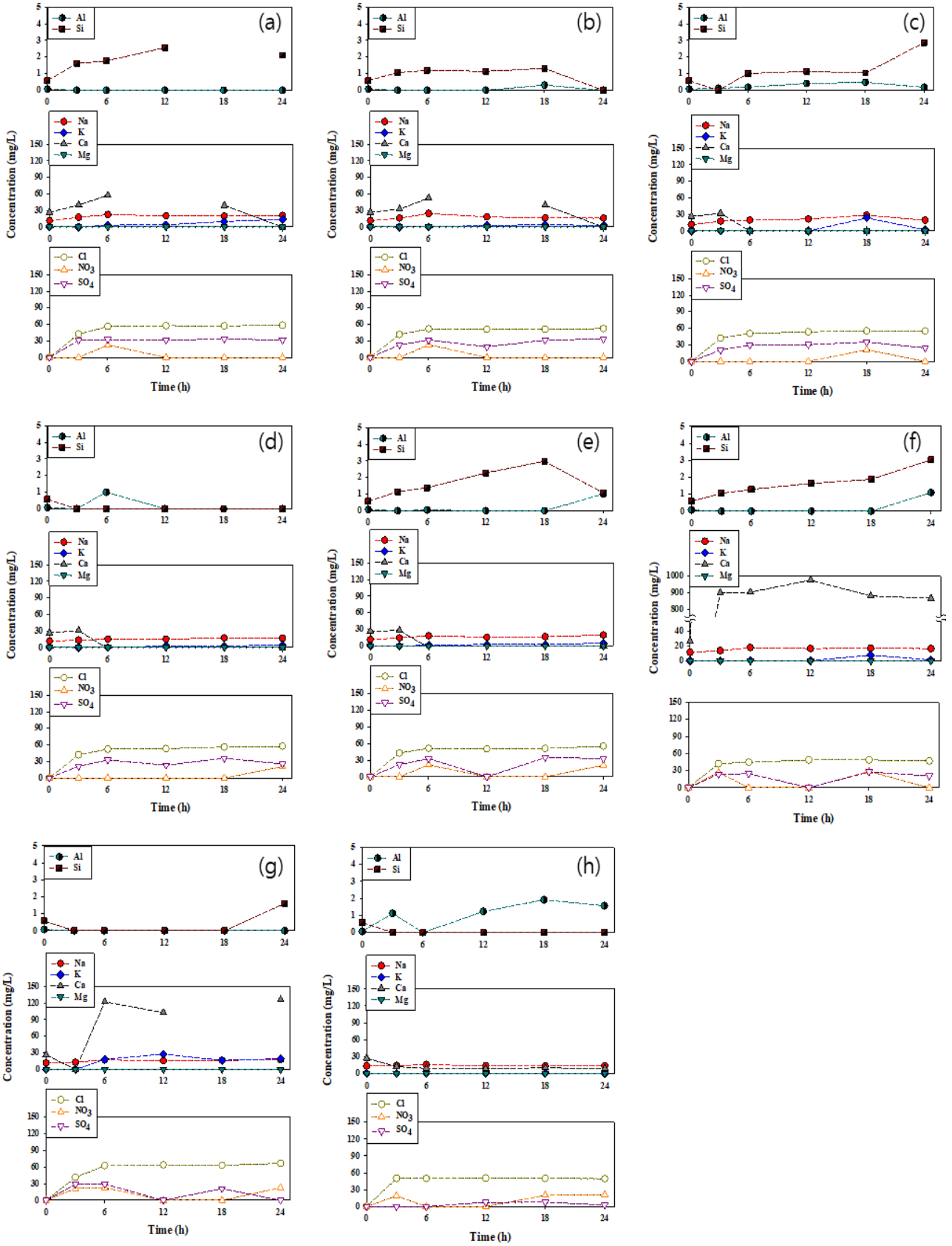

Fig. 1 Dissolution kinetics of dissolved elements and ions in different mineral suspensions; (a) feldspar, (b) olivine, (c) elvan, (d) red clay, (e) illite, (f) lime, (g) sericite, and (h) zeolite. Experimental condition: mineral concentration = 10 g/L in 500 of tap water, mixing rate = 150 rpm, mixing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

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

Table 3 Calculated RMSE and Chi-Squared ($\chi 2$) Values between the Experimental Data and Model Calculations (Shown in Fig. 1) for the phosphorus and pH changes associated with lime/mineral dissolution[a]

adsorption	material	RMSE	$\chi 2$	$\chi c2$
individual	phosphorus	0.10	0.15	3.84
	pH changes	0.09	0.43	
simultaneous	phosphorus	0.05	0.13	6.47
	pH changes	0.07	0.59	

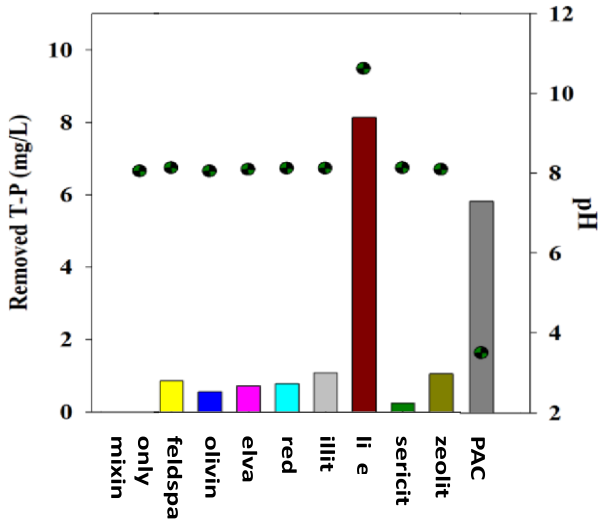

Fig. 2 shows the total amount of T-P concentration

Ai, 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 Ca^{2+} (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 Ca^{2+} rich solution with several hundred mg/L concentration. This is because that lime reaction with water can form calcium hydroxide ($CaO + H_2O \rightarrow Ca(OH)_2$) and the calcium hydroxide can be dissolved in water (i.e., $Ca(OH)^2 \rightarrow Ca^{2+} + 2\ OH^-$, solubility in water = 1.73 g/L at 20 C°), producing an alkaline solution higher than pH 12.5 (Rumble 2018).

In the process of utilizing lime/mineral dissolved solution for the treatment of phosphate-containing wastewater, the corresponding pH changes associated with lime/mineral dissolution are crucial. As shown, the model accurately reproduces the experimental data (see the low RMSE and chi-squared values in Table 3).

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

Fig. 2 T-P removal in wastewater by various mineral/ lime dissolved solutions and a commercial coagulant (i.e., PAC). Experimental condition: Volume of waste- water = 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 Al_2O_3), mixing at 150 rpm for 5 min and slow mixing at 20 rpm for 20 min removed in real

Table 4 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

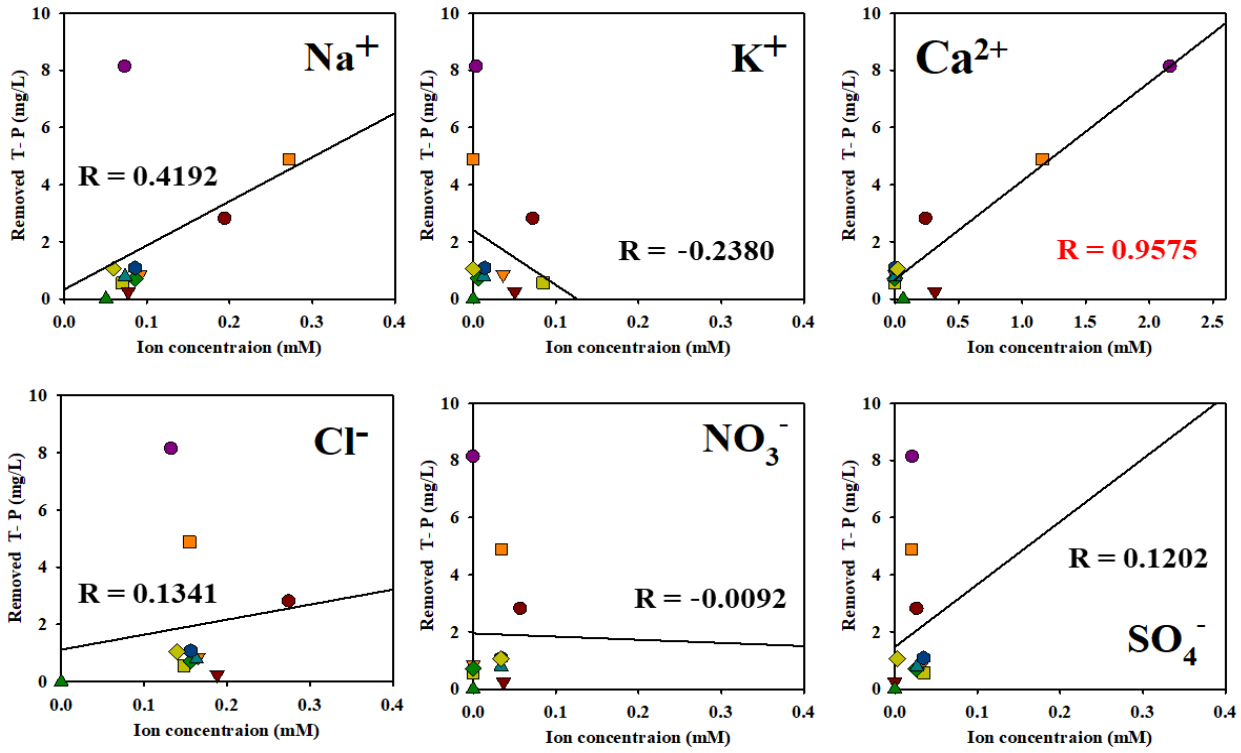

Fig. 3 Co-relationship between the T-P removal amount and each ion concentration

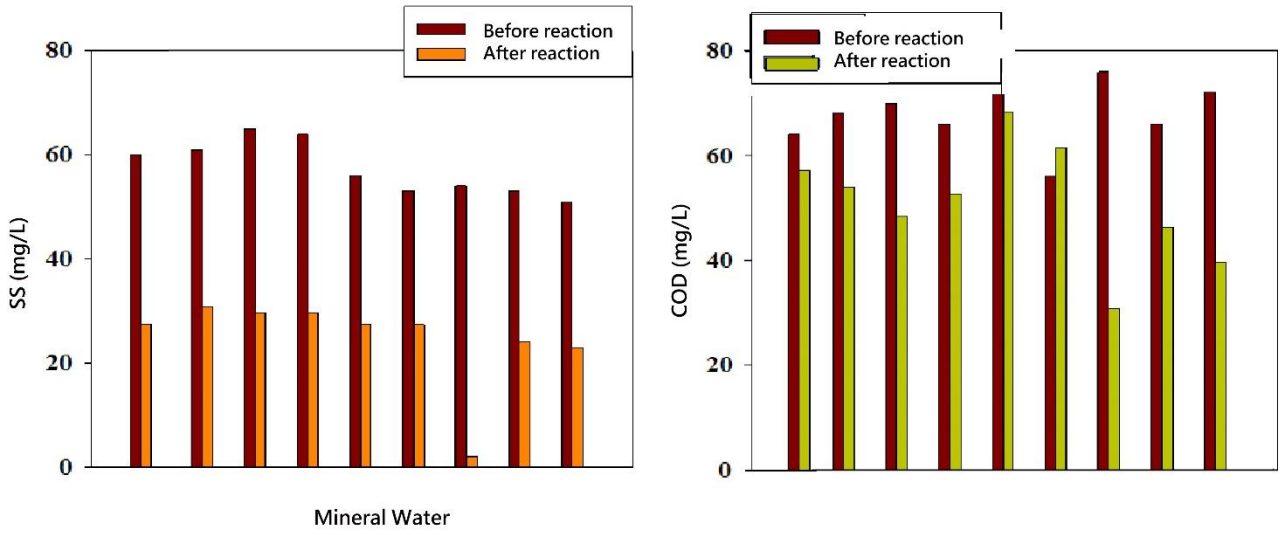

Fig. 4 Removal of (a) SS and (b) COD by various mineral/lime dissolved solutions. Experimental condition: Volume of wastewater = 275 mL, Volume of added mineral/lime solution (10% diluted solution) = 25 mL, mixing at 150 rpm

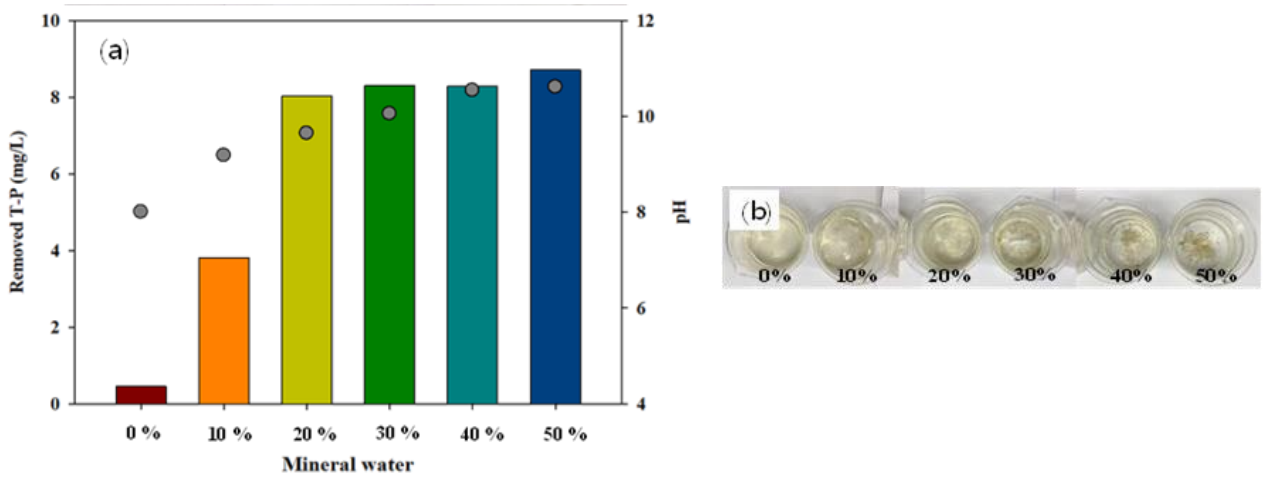

Fig. 5 T-P removal in wastewater by lime-feldspar dissolved solutions prepared in different ratio. Experimental condition: Volume of wastewater = 450 mL, [T-P]0 = 8.76 ~9.48 mg/L, Volume of added solution (10% diluted solution) = 50 mL, mixing at 150 rpm for 5 min and slow mixing at 20 rpm for 20 min

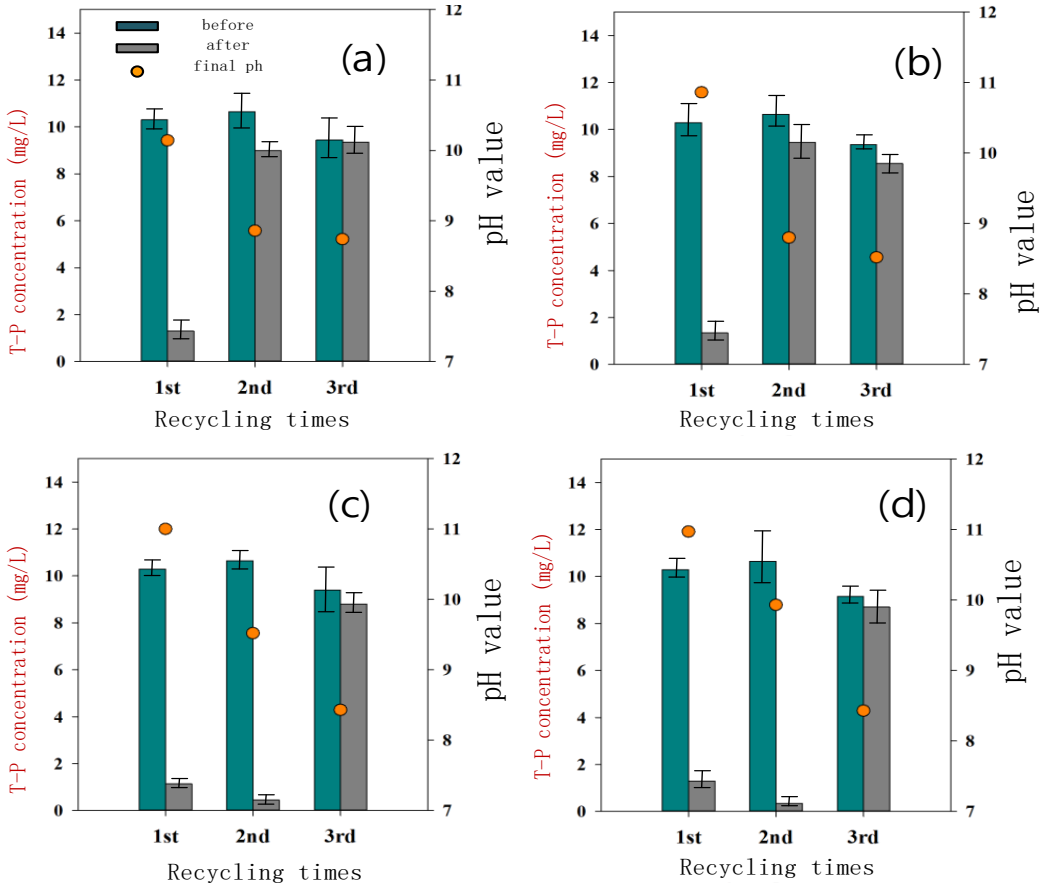

Fig. 6 Successive T-P removal in wastewater by lime-feldspar dissolved solutions prepared in different ratio ((a) 20, (b) 30, (c) 40, and (d) 50%). Experimental condition: Volume of wastewater = 450 mL, [T-P]0 = ~10 mg/L, Volume of added solution (10% diluted solution) = 50 mL, mixing at 150 rpm for 5 min and slow mixing at 20 rpm for 20 min

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 HPO_4^- or PO_3^{4-} and these P species can react with the dissolved Ca^{2+} in lime dissolved solution. The high pH could improve the Ca-P precipitation reaction to form hydro- xyapatite-like mineral ($Ca_5(PO_4)_3(OH)$ or $Ca_{10}(PO_4)_6(OH)_2$) (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 Ca^{2+} ions, whereas other ions did not show any co-relationship in this study. The result indicate that the Ca^{2+} 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 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 Ca^{2+} 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 Ca^{2+} 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.

Fig. 5(b) shows that the amount of precipitate also increased at higher ratio of lime to feldspar.

During the process of lime and minerals dissolution, the concentration of Ca^{2+} typically exhibits initial ascending trends followed by subsequent declines. This may suggest the formation of insoluble substances, such as CaCO3 during the process.

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 overcame by higher ratio of lime content in this study. However, there is no clear co-relationship between the total Ca^{2+} 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.

4. Conclusions

In previous studies, only iron (Fe3+) and calcium (Ca2+) were commonly used to remove PO4-P (considered T-P in this study) from wastewater through chemical precipitation. Here, various minerals and dissolved lime solutions are used to remove total phosphorus (T-P) from the wastewater. The dissolution kinetics of different minerals (feldspar, olivine, mafic, illite, sericite and zeolite) were compared and lime and its solutions were used for T-P removal from real wastewater. Lime solution has the highest total phosphorus removal rate (about 90%), followed by zeolite, illite, feldspar, etc. We observed a significant correlation between the initial amount of Ca2+ and T-P elimination (R = 0.96). The peak of lime (CaO) began to appear in high temperature, and the calcite (CaCO3) content decreased significantly. As the thermal activation temperature increased, the lime (CaO) content further increased and the calcite (CaCO3) content decreased. This is due to the formation of hydroxyapatite minerals through the Ca-P precipitation reaction in high pH solutions. In addition, suspended solids (SS) and chemical oxygen demand (COD) are also removed simply by dissolving the lime solution. Finally, lime solution and feldspar dissolution were prepared in different proportions (10-50%) and the results showed that 40% and 50% of the samples produced T-P twice in a row. Lime in water treatment does not appear to represent a problem with potential contaminant release. If concentrations of specific chemicals are elevated in the source water, the concentrations of these chemicals in the lime may also be elevated, and samples should be taken for comparison before reusing the material with the minerals. Besides Ca2+, certain cations (such as Fe3+, Al3+, Mg2+, etc.) may form phosphate precipitates or hydroxide flocs for phosphate removal. Therefore, if the mineral-dissolved solution is involved in the treatment of phosphate-containing wastewater, it is important to provide a comprehensive discussion on the effectiveness and mechanism of these cations, and which would be further parts in the futuristic research. Furthermore, in real wastewater, total phosphorus consists not only of PO43-. Therefore, it is necessary to include discussions on the mechanisms for the removal of organic phosphorus and probably non-orthophosphate species. The quantitative and qualitative analysis of the solid products has not been included in this manuscript, while these data are essential for elucidating the entire process.

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