

# Phosphorus desorption and recovery from aqueous solution using amorphous zirconium hydroxide/MgFe layered double hydroxides composite

*by* Atin Nuryadin

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## Phosphorus desorption and recovery from aqueous solution using amorphous zirconium hydroxide/MgFe layered double hydroxides composite

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**Abstract.** Global phosphorus scarcity implies the importance of phosphorus recovery. Desorption is an essential process in phosphate removal by adsorption technique by enabling two crucial aspects: the reusability of adsorbent and the recovery of phosphorus. In this study, phosphate desorption by NaOH for composite reusability and phosphorus recovery by CaCl<sub>2</sub> were investigated. Based on the cost analysis, the uncalcined amorphous zirconium hydroxide/MgFe layered double hydroxides composite (am-Zr/MgFe-LDH) with Zr to Fe molar ratio of 1.5 was effective in reducing cost for phosphate adsorption compared to amorphous zirconium hydroxide (am-Zr) and MgFe layered double hydroxide (LDH). The XRD analysis indicated that phosphate desorption was preferably performed by stripping adsorbed phosphate on the composite surface using NaOH solution. The reuse of 2 N NaOH for composite regeneration could effectively maintain a higher adsorption ability (86%) than 1 N NaOH, and additionally, could be considered as an economic regeneration agent. The composite was chemically stable in maintaining its structure during eight adsorption-desorption cycles. The mechanisms involved during phosphate desorption by NaOH were mainly ligand exchange and electrostatic repulsion. The phosphorus recovery showed that the optimum recovery (~95%) was obtained by adding CaCl<sub>2</sub> at pH 13 and calcium to phosphorus molar ratio of 3.5.

### 1. Introduction

The release of phosphate-containing wastewater into water bodies results in serious eutrophication problems and the annual loss of usable phosphorus [1,2]. Phosphorus is one of the fundamental elements for all living organisms and is globally used in food production [3]. Agriculture is still the dominant user of global phosphorus, accounting for about 80% of the total annually mined phosphorus deposits mainly used to produce agricultural fertilizers [4]. To secure food production sustainability in the future, the demand for these fertilizers is expected to increase continuously since the population is projected to grow by about 1.9 billion inhabitants from 2020 to 2050 [5]. The world's commercial phosphorus fertilizers are mainly produced from mined phosphorus rock, which is a non-renewable, limited, and geographically nonhomogeneous resource [6]. These conditions imply the essential of dealing with not only phosphate removal but also phosphorus recovery and reuse.



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Phosphorus recovery from wastewater has been considered an effective strategy to regenerate this non-renewable resource and conserve the water environment quality [7]. Chemical precipitation of phosphate into calcium phosphate has become an attractive method for phosphorus recovery from wastewater since calcium phosphate is recognized as an agricultural fertilizer with a low solubility [8]. However, the coexistence of other metal ions commonly found in sewage, such as  $Mg^{2+}$  and  $Zn^{2+}$ , has been found to interfere with the precipitation of calcium phosphate [9]. Consequently, these interfering ions need to be removed from the water before the precipitation process. Adsorption has become a fascinating separation technique of phosphate from water because of the possibility of phosphorus recovery [10]. The phosphorus recovery is performed by precipitation of desorbed phosphate in the desorption solution obtained from the adsorbent regeneration. The selective phosphate adsorption by an adsorbent can decrease the coexistence of other anions in the desorption solution [11]. As a result, the interference of unwanted anions during the phosphate precipitation process can be minimized and subsequently increase the effectiveness of phosphate recovery.

In the previous study, a composite of amorphous zirconium hydroxide/MgFe layered double hydroxides (am-Zr/MgFe-LDH) was synthesized by a novel two-stage synthesis. A series of batch and column tests revealed that the uncalcined am-Zr/MgFe-LDH with a Zr:Fe ratio of 1:5 had a high phosphate and maintained high removal efficiency over eight adsorption-desorption cycles by NaOH solution [12,13]. However, a comprehensive understanding of the phosphate adsorption mechanism by am-Zr/MgFe-LDH is essential to consider an appropriate reagent for composite regeneration, such as the NaOH solution used in the previous study. In addition, the reusability of an adsorbent needs technical and financial efforts for its practical application possibility [14]. The feasibility of the am-Zr/MgFe-LDH regeneration has been only studied technically. Therefore, it is essential to perform a further investigation on the economic feasibility of the composite reusability. The purpose of this study was to analyze the cost-effectiveness of am-Zr/MgFe-LDH synthesis and its regeneration as a phosphate adsorbent. The suitability of NaOH as a desorption agent was investigated through adsorption mechanism analysis, and then the desorption mechanism was proposed. An alternative desorption agent was examined to decrease the cost of composite reuse further. In addition, the recovery of phosphorus from desorption solution obtained from am-Zr/MgFe-LDH regeneration was evaluated.

## 2. Materials and Methods

### 2.1. Preparation of adsorbent

All the chemicals used for the am-Zr/MgFe-LDH preparation were analytical pure-grade reagents acquired from FUJIFILM Wako Pure Chemical Corporation. The composite with a Zr to Fe molar ratio of 1.5 was prepared by a two-stage coprecipitation method previously established [12].

### 2.2. Characterization of adsorbent

X-ray diffraction (XRD) analysis was carried out to obtain the crystalline structure of the am-Zr/MgFe-LDH. The XRD pattern was obtained using a Rigaku Ultima IV Protectus diffractometer equipped with a monochromator  $CuK\alpha$  source at 40 kV. The recording was carried out with a scanning rate of  $1^\circ\text{min}^{-1}$  in the  $2\theta$  angle range from  $5^\circ$  to  $70^\circ$ .

### 2.3. Phosphate desorption and adsorbent reusability

The desorption of phosphate from composite was investigated after the adsorption process of 0.1 g am-Zr/MgFe-LDH in  $10\text{ mg-P L}^{-1}$  phosphate solution. The phosphate concentration in the filtrate after the adsorption process was measured by UV-vis spectrophotometry. Phosphate desorption was performed by reusing the same 2 N NaOH during eight adsorption-desorption cycles. The dried P-loaded composite was subsequently added to NaOH solution with a dose of 2 mg/L and shaken at 150 rpm. After 40 min, the phosphate concentration in the supernatants was measured, and the desorption efficiency was determined by equation (1):

$$DE (\%) = \frac{CV}{qm} \times 100 \quad (1)$$

where  $C$  is the phosphate concentration after the desorption process ( $\text{mg-P L}^{-1}$ ),  $V$  is the volume of solution (L),  $q$  is the amount of phosphate adsorbed on the composite ( $\text{mg-P g}^{-1}$ ), and  $m$  is the weight of adsorbent in desorption experiment (g). The composite reusability was examined by subsequently reusing the previously desorbed composite through an eight-cycle of adsorption-desorption and calculating the adsorption performance at the end of each cycle.

#### 2.4. Phosphorus precipitation

The precipitation performance study was investigated using regenerated desorption solution (reused 2N NaOH) after eight operation cycles. The desorbed phosphate was precipitated by adding 0.1 M of  $\text{CaCl}_2$  in the phosphate-containing NaOH solution. To investigate the effect of calcium to phosphorus (Ca/P) molar ratio on the phosphorus precipitation, the  $\text{CaCl}_2$  solution was added to obtain different Ca/P molar ratios of 0.5–4. The effect of pH on the precipitation was studied by adjusting solution pH to 9–13. After the precipitation process, the concentration of residual phosphate on the filtrate was measured to determine the precipitation rate using equation (2):

$$PR(\%) = \frac{C_0 - C}{C_0} \times 100 \quad (2)$$

where  $C$  and  $C_0$  are the initial and residual phosphate concentrations ( $\text{mg-P L}^{-1}$ ), respectively. After phosphate measurement, the precipitate was calcined at  $500^\circ\text{C}$  and analyzed using XRD analysis.

#### 2.5. Cost estimation

The cost of the phosphate removal process mainly depends on the adsorbent cost. This study estimated the cost of phosphate adsorption and adsorbent regeneration using the raw material prices for adsorbent synthesis and regeneration agent, respectively. In addition, the energy cost was involved in the cost estimation of calcined adsorbents. The electricity cost per kWh used is based on Japan's average electricity price for 2020 ( $\sim 26.00$  JPY) as acquired from TEPCO Energy Partner, Inc. [15].

The costs calculated in this study are all in the Japanese Yen (JPY). The chemical cost  $CC$  (JPY) of adsorbent required for phosphate adsorption was calculated from the total chemical cost used for adsorbent synthesis using equation (3):

$$CC = \sum \left( \frac{m_s}{m_o} \times m_u \times CP \right) \quad (3)$$

where  $m_s$ ,  $m_o$ , and  $m_u$  are the weight of a chemical used in synthesis, obtained composite after synthesis, and composite used in adsorption (g), respectively, and  $CP$  is the chemical price (JPY/g). The energy cost  $EC$  (JPY) was added to the total cost of calcined samples, which was calculated by equation (4):

$$EC = P \times a \times t \times EP \quad (4)$$

where  $P$  is the power consumed by the furnace (kW),  $a$  is a load factor ( $a = 1$  if the furnace is used in full mode),  $t$  is the duration of the calcination process, and  $EP$  is the energy price (JPY/kWh). Subsequently, the cost for phosphate adsorption (JPY/mg-P) was calculated by equation (5):

$$\text{Cost for phosphate adsorption} = \frac{q}{CC + EC} \quad (5)$$

where  $q$  is the amount of adsorbed phosphate ( $\text{mg-P/g}$ ). The adsorbent regeneration cost was estimated from the cost of NaOH consumed in the desorption process. The desorption cost  $DC$  (JPY) was calculated using equation (6):

$$DC = V \times M \times M_r \times CP \quad (6)$$

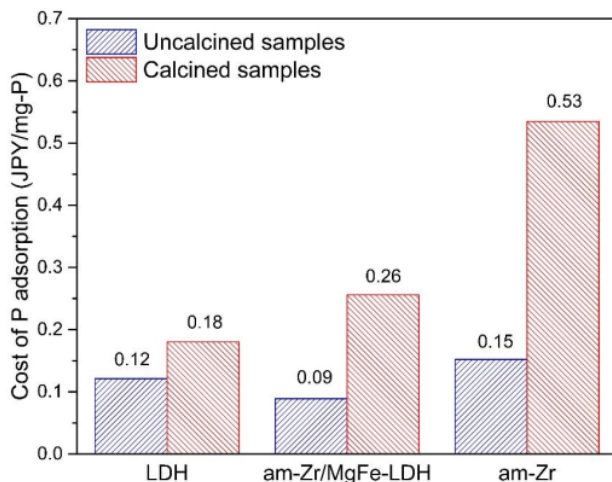
where  $V$  is the volume of NaOH solution used for desorption (L),  $M$  is the molarity of NaOH (mol/L), and  $M_r$  is the molecular weight of NaOH (g/mol).

### 3. Results and Discussion

#### 3.1 Analysis of cost for phosphate adsorption

Cost estimation is essential in determining the applicability of an adsorbent for practical applications. In the previous study, it was found that the adsorption capacity of layered double hydroxides (LDH) could increase by either calcining it or introducing amorphous zirconium hydroxide (am-Zr) to the LDH matrix (as am-Zr/MgFe-LDH) without calcination [12]. Though the adsorption ability of uncalcined am-Zr/MgFe-LDH was obtained to be higher than both LDH and am-Zr, it was necessary to estimate further the cost of adsorbents application to investigate their economic feasibility.

Figure 1 compares the feasibility of adsorbents, with and without calcination, to economically adsorb phosphate based on the adsorption capacity obtained from the batch studies. The figure shows the cost estimation of each adsorbent required to remove 1 mg-P of phosphate from a solution. The cost estimation was derived from the use of chemicals and energy in the calcination process during adsorbent preparation. The figure specifies that the cost associated with the uncalcined am-Zr/MgFe-LDH was found to be less than other studied adsorbents. The price for removing phosphate by am-Zr/LDH was about half less than the calcined LDH. The elimination of the calcination process during the LDH synthesis by combining it with am-Zr can produce an economically feasible adsorbent. Moreover, the costs for phosphate removal by uncalcined composites are lower than am-Zr, indicating that the price of fully am-Zr usage as an adsorbent can be reduced by composite synthesis.



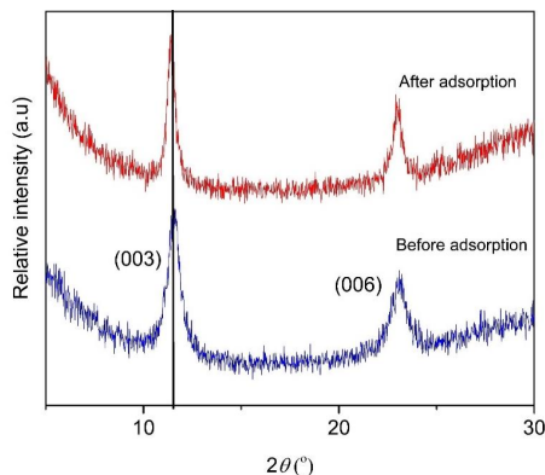
**Figure 1.** Comparative cost for the removal of 1 mg-P of phosphate using studied adsorbents.

#### 3.2 Analysis of method for desorption

In the XRD pattern of LDH, the intercalation of phosphate anions into the LDH interlayer is characterized by the shift (003) reflection to a lower  $2\theta$  angle, corresponding to the enlargement of the basal distance  $d_{003}$  compared to the host LDH [16]. The XRD pattern of am-Zr/MgFe-LDH in the  $2\theta$  range of  $15\text{--}30^\circ$  is presented in Figure 2. It is shown that no obvious displacement on the (003) diffraction plane was observed after the phosphate adsorption process suggesting that the anion exchange is not the primary mechanism during the phosphate adsorption process. Most phosphates were adsorbed on the composite surface through surface adsorption, both electrostatic attraction and inner-sphere complexation than via anion exchange [13]. This implied that the desorption process was better

performed by stripping adsorbed phosphate on the adsorbent surface rather than exchanging phosphate intercalated in the LDH interlayer.

Moreover, regenerating composite by  $\text{NaCO}_3$  solution, to replace back phosphate anion in the LDH interlayer by carbonate anion, can negatively affect subsequent phosphate adsorption. As previously revealed that carbonate anion is the most competitive anion for phosphate adsorption onto the composite surface. On the other hand, the carbonate anion on the desorption solution makes it difficult to recover phosphorus as calcium phosphate because of the calcium carbonate formation [17].



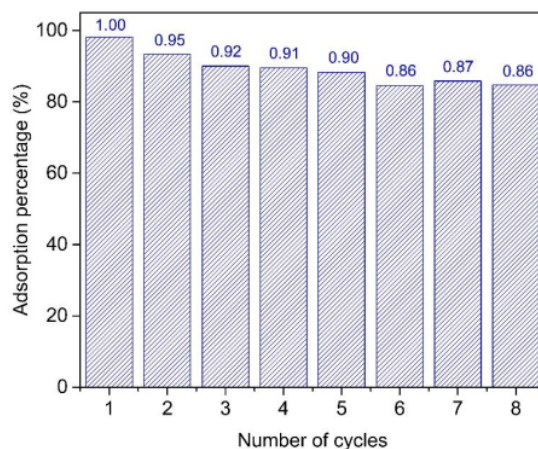
**Figure 2.** X-ray powder diffraction patterns of am-Zr/MgFe-LDH before and after adsorption

### 3.3 Adsorbent reusability

The preceding study showed that am-Zr/MgFe-LDH regenerated by 1 N NaOH could maintain 92% of its original adsorption efficiency after four regeneration cycles and then continued to decrease to 78% after seven cycles [12]. To find the adequate number of regeneration cycles by 1 N NaOH, the estimation cost for one cycle was compared with the cost for synthesizing fresh am-Zr/MgFe-LDH with the same weight as that used in regeneration. The comparison showed that a reusability cycle by 1 N NaOH only reduced ~9% of the new composite synthesis with a 5% performance decrease. Therefore, a further increase in the number of cycles is insufficient to reduce the cost of synthesizing the new composite.

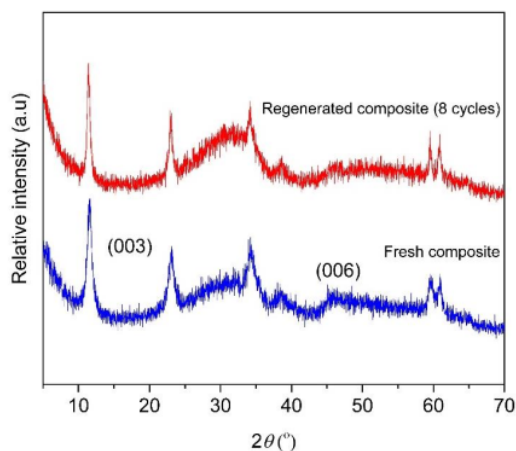
In this study, to find an economical method for the adsorbent regeneration, the application of reused 2 N NaOH (using the same NaOH solution for each desorption cycle) was also investigated in the reusability experiments. Figure 3 displays the reusability of the composite during eight cycles by reused 2 N NaOH. The numbers above the bar on the figure are adsorption retain ratio of phosphate adsorption by the composite. After the first cycle, the composite retained 95% of the adsorption ability of the new composite. Subsequently, its adsorption ability gradually declined continuously to 86% after the eighth regeneration cycle. The result of the reusability study demonstrated that the composite desorbed by reused 2 N NaOH in the desorption process could hold a higher adsorption efficiency than that desorbed by 1 N NaOH. A high removal efficiency retention of composite over eight regeneration cycles by reused 2 N NaOH emphasizes the stability of the composite during the regeneration process.

The economic feasibility of the reused 2 N NaOH application was also investigated as in the regeneration by 1 N NaOH. The cost estimation calculation showed that the application of reused 2 N NaOH during eight cycles cost ~4.4 times lower than the eight-time synthesis of the new composite. In addition to maintaining a higher adsorption capacity during eight cycles, this finding also indicated that reused 2 N NaOH solution could be considered an economic regeneration agent for the reuse of am-Zr/MgFe-LDH.



**Figure 3.** The reusability of composite, which regenerated by reused 2 N NaOH.

The cost-effectiveness of an adsorbent depends on its reusability. Consequently, adsorbents with high chemical stability are highly attractive due to their strength during the regeneration process by an extremely high acidic or alkaline desorption agent. The XRD pattern 8-cycle regenerated am-Zr/MgFe-LDH is presented in Figure 4. The figure compares it with the XRD pattern of fresh synthesized am-Zr/MgFe-LDH to determine the stability of the composite structure. It is shown that the peaks of the fresh composite were still maintained even after eight adsorption-desorption cycles using 2 N NaOH. This result clearly emphasized the chemical stability of am-Zr/MgFe-LDH in high alkaline conditions.



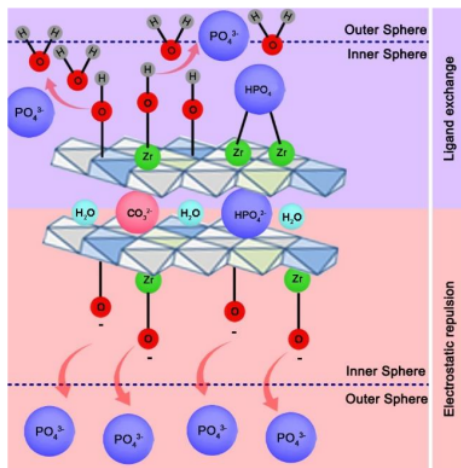
**Figure 4.** X-ray powder diffraction patterns of fresh and 8-cycle regenerated am-Zr/MgFe-LDH.

### 3.4 Desorption mechanism

As adsorption mechanisms, the fundamental of desorption mechanisms is crucial for consideration in applying an adsorbent in full-scale phosphate adsorption [18]. Desorption of adsorbed phosphate from the composite by NaOH solution can be described as the reverse process of the phosphate uptake by surface adsorption, as shown in Figure 5. High NaOH concentration and low phosphate concentration promote the phosphate desorption process by ligand exchange between adsorbed phosphate species and



hydroxyl groups [11]. Furthermore, a high concentration of  $\text{OH}^-$  results in the deprotonation of hydroxyl groups in the composite surface [19]. This reaction causes a negatively charged composite surface, which creates the repulsion of phosphate adsorbed by electrostatic attraction and subsequently shields phosphate re-adsorption during the desorption process.



**Figure 5.** Proposed mechanism of phosphate desorption from am-Zr/MgFe-LDH by NaOH solution.

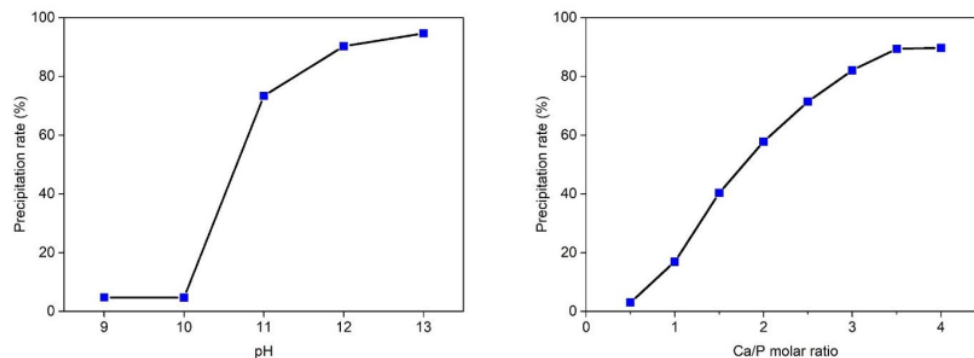
Desorption experiment results showed that the phosphate adsorption process was not fully reversible. The loss of efficiency might be attributed to the strongly bound phosphates with metal oxyhydroxides on the composite surface and other interfering parameters, such as the partial destruction of adsorption sites during the regeneration process. Electrostatically bound phosphates are easier to desorb than inner-sphere complex bound phosphates. The irreversible phosphates mostly come from inner-sphere complex bound phosphates, which are bound by bidentate ligands. Bidentate phosphates are firmly attached to the composite surface because they are chemically bound by two bonds, compared to monodentate phosphates that are linked by a single bond.

### 3.5 Precipitation of desorbed phosphate

The major problem of the regeneration process by 2 N NaOH in the actual wastewater treatment plant is the disposal of the high alkaline desorption solution, which contains a high concentration of phosphate. To comply with pH discharge limits, which range between 6 and 9, wastewater treatment plants must implement a wastewater pre-treatment [20]. The precipitation of phosphate to hydroxyapatite (HAP) was proposed to be the one of the effective methods to tackle this issue. The HAP has been shown to be effective as phosphate fertilizer for agriculture [11]. Du et al. (2013) precipitated HAP by adding  $\text{CaCl}_2$  to a phosphate solution at pH 10–11 [21]. Before the precipitation process, the desorption solution pH (~14) needs to be reduced by dosing acid until the typical pH required for phosphate precipitation. The HAP precipitation by  $\text{CaCl}_2$  is followed by the decrease of pH solution to the range of discharge standard ( $6 < \text{pH} < 9$ ) [22]. In this way, the pH of the desorption solution can be reduced, and the phosphate can be recovered to valuable compounds simultaneously.

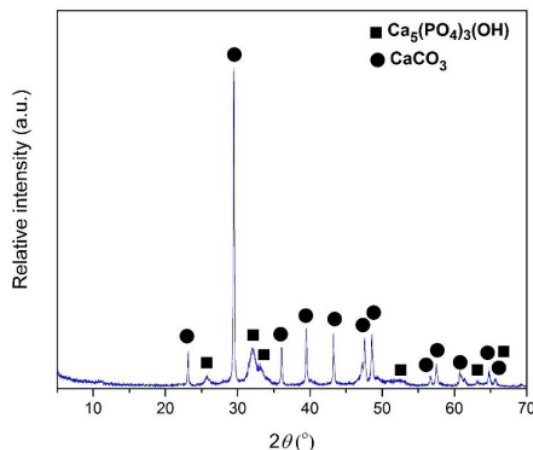
To recover phosphate from desorption solution after the regeneration process, the precipitation of HAP by  $\text{CaCl}_2$  was carried out in this study. The solution pH and calcium to phosphorus (Ca/P) are crucial parameters that determine the phosphate precipitation [23]. Therefore, it is essential to investigate the effect of solution pH and Ca/P molar ratio on the phosphate precipitation rate. The results are shown in Figure 6. The figure reveals that the measured residual phosphate concentration in the regeneration solution is a function of Ca/P molar ratio and solution pH. Figure 6(a) exhibits that Ca/P

molar ratio of  $\sim 3.5$  was obtained to be the effective ratio to achieve a nearly complete transformation of P into HAP. This result indicated that the excess Ca was preferable to precipitate phosphate as compared with the theoretical Ca/P of HAP (1.67) [7]. In addition, the phosphate precipitation was obtained higher at pH 13 than other observed pH values, as illustrated in Figure 6(b).



**Figure 6.** The effect of Ca/P molar ratio at pH 12 (a) and pH at Ca/P molar ratio of 3.5 (b) on the precipitation of calcium phosphate.

The XRD pattern analysis was performed to examine the structure of the recovered phosphate product, as illustrated in Figure 7. The XRD analysis showed that the peaks observed on the XRD pattern matched HAP ( $\text{Ca}_5(\text{PO}_4)_3(\text{OH})$ ) and calcium carbonate ( $\text{CaCO}_3$ ) characteristic peaks. This result implied that the formed precipitate under the experimental conditions in this study was HAP with  $\text{CaCO}_3$  as a by-product. The coexistence of  $\text{CaCO}_3$  was due to the  $\text{Ca}^{2+}$  that readily reacts with hydroxyl ions at high pH to form  $\text{Ca}(\text{OH})_2$  and forms  $\text{CaCO}_3$  by subsequently reacting with dissolved  $\text{CO}_2$  [9].



**Figure 7.** The X-ray powder diffraction pattern of the phosphate precipitation product.

#### 4. Conclusion

The combination of am-Zr and LDH as am-Zr/MgFe-LDH produced a low-cost phosphate adsorbent with a high adsorption capacity. Phosphate desorption is essential for phosphate adsorbents since the cost-effectiveness of an adsorbent depends on its reusability. The desorption of adsorbed phosphate on the composite surface by alkaline solution was found to be an appropriate method for am-Zr/MgFe-

LDH regeneration. The eight consecutive regeneration cycles that were carried out indicated that the composite could maintain its structure and high adsorption ability, showing the potential reusability of the composite for phosphate adsorbent. It was proposed that the phosphate desorption mechanisms by NaOH solution were predominantly ligand exchange and electrostatic repulsion. This study elucidates a new economical method for am-Zr/MgFe-LDH regeneration by reusing 2 N NaOH in each desorption process which can be considered in practical application. The desorbed phosphate from the composite was successfully recovered as HAP, which can be applied as agricultural fertilizer. The results indicated that the application of am-Zr/MgFe-LDH for phosphate removal from water has high chemical stability during the regeneration process and excellent potential for phosphate recovery as HAP.

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