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Development of a Novel Phosphate Ion Adsorbent Using Calcined Fossil Coral

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Phosphorus recovery using adsorption methods has the potential to address both the problem of phosphorus accumulation in aquatic environments and the shortage of phosphorus resources. Fossil corals (FCs), which are the skeletal remains of dead corals composed of calcium carbonate, have potential as phosphorus adsorbents. In this study, we investigated the processing conditions for FCs that were optimized for phosphorus recovery. The main component of FCs is calcium carbonate (calcite), which thermally decomposes at approximately above 800°C to form calcium oxide. The specific surface area decreased with increasing temperature up to 800°C and then increased significantly at 900–1000°C. The amount of phosphate ions adsorbed was the highest for FCs calcined at 1000°C and lowest for FCs calcined at 800°C, showing a trend similar to that of the specific surface area. After phosphate adsorption, brushite was formed on all adsorbents, and hydroxyapatite additionally formed on FC900 and FC1000. The amounts of phosphate ions adsorbed by FC–FC800 showed a decreasing trend with increasing initial pH, whereas FC1000 exhibited the opposite trend. These results suggest that the main recovery mechanism of FC–FC800 is electrostatic adsorption, while that of FC1000 is calcium phosphate precipitation, and that of FC900 involves both mechanisms. These findings demonstrate that FCs are promising raw materials for phosphorus recovery and for sustainable recycling as a calcium phosphate resource.

Key words fossil coral, water environment, phosphate, adsorption, calcium

INTRODUCTION

Phosphorus is indispensable for synthetic fertilizers, chemical product manufacturing, and metal processing,¹⁾ though phosphate ore supplies, on which Japan depends almost entirely through imports, will be depleted worldwide in the coming century.²⁾ On the other hand, phosphorus accumulation in aquatic environments is caused by inflow of industrial wastewater and has reportedly increased by 75% in freshwater since the Industrial Revolution.³⁾ This phosphorus accumulation causes eutrophication, resulting in explosive algae blooms that adversely affect fisheries and drinking water.⁴⁾ Therefore, although phosphorus supply is insufficient, substantial amounts of phosphorus are present in the environment. Recovering and recycling phosphorus from aquatic environments can provide a solution to phosphorus problems from both environmental and resource perspectives.

The methods available for phosphorus recovery include adsorption,⁵⁾ chemical precipitation,⁶⁾ biological treatment,⁷⁾ and electrocoagulation.⁸⁾ Among these methods, adsorption

offers advantages such as easy handling, compact equipment, and low labor costs.⁹⁾ Recently, phosphorus adsorbents have been developed from wastes containing iron,¹⁰⁾ magnesium,¹¹⁾ and calcium,¹²⁾ which are used in lakes and rivers for detention.¹³⁾ Our laboratory has developed a phosphorus adsorbent from oyster shells,¹²⁾ in which phosphate ions reacted with calcium carbonate (the principal component) or calcium oxide (the main component after calcination) to form hydroxyapatite (HAp). The resulting HAp could be used as fertilizer,¹⁴⁾ which allows for the recycling of phosphorus resources. Oyster supply stability is a concern because the production volume fluctuates seasonally.¹⁵⁾ Therefore, alternative resources must be identified to ensure a stable supply of adsorbents.

Fossil corals (FCs) are the skeletal remains of dead corals and are composed primarily of calcium carbonate.¹⁶⁾ Coral mortality has increased in recent years owing to global warming,¹⁷⁾ and a similar trend has been observed in Japan.¹⁸⁾ There are concerns that the growing abundance of FCs could negatively affect ecosystems and biodiversity,¹⁹⁾ highlighting the need to develop sustainable utilization strategies. The min-

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eral composition of FCs varies with habitat,²⁰⁾ and the crystals of their calcium carbonate compounds also vary in form.²¹⁾ The crystal form of calcium carbonate influences phosphorus adsorption, with calcite exhibiting greater adsorption capacity than aragonite. Upon calcination at 400°C, aragonite is transformed into calcite, which is highly thermally stable, and at $\geq 900^\circ\text{C}$, calcium carbonate decomposes to calcium oxide.¹⁶⁾ These characteristics suggest that FCs can adsorb phosphorus via multiple adsorption mechanisms, including HAp formation, similar to calcined oyster shells.

To address the abovementioned gaps, we investigated the processing conditions for FCs optimized for phosphorus recovery and elucidated the adsorption mechanism, with the aim of recycling phosphorus resources.

MATERIALS AND METHODS

Materials and Chemicals Fossil corals (FCs) were purchased from CORAL INTERNATIONAL INC. (Okinawa, Japan). The materials were dried at 50°C for 72 h and then crushed until the particle size was less than 500 μm , and the sample was named FC. A muffle furnace was used to calcine FC. The temperature was increased from room temperature to 200, 400, 600, 800, 900, and 1000°C over 2 h and then maintained at each target temperature for an additional 2 h under a nitrogen atmosphere. The resulting samples were labeled FC200, FC400, etc., corresponding to their respective calcination temperatures. Potassium dihydrogen phosphate was used as the adsorbate, and HCl and NaOH were used for pH adjustment. All reagents were purchased from FUJIFILM Wako Pure Chemical Corporation (Osaka, Japan). Pure water was used in all the experimental systems.

Characterization of Each FC The physicochemical properties of each FC were evaluated as described below. The surface microstructure of FC was assessed using scanning electron microscopy (SEM; Regulus8240, Hitachi High-Technologies Corporation, Tokyo, Japan), with the instrument operated at an acceleration voltage of 10.0 keV. Changes in the physical properties of FC during heating were evaluated using thermogravimetric-differential thermal analysis (TG-DTA; TG8210, Rigaku, Tokyo, Japan) operated at target temperature of 1000°C, heating time of 10°C/min, atmosphere of 200 mL/min, and sampling time of 1 s. Powder X-ray diffraction (XRD; MiniFlex II Rigaku Corporation, Tokyo, Japan) was used for the crystal structure analysis of FC, operated at a radiation source of CuK α , tube voltage of 30 kV, and tube current of 15 mA. Meanwhile, the specific surface area of each FC was measured using a BELSORP MINI X instrument (MicrotracBEL, Osaka, Japan). The zero-charge point (pH_{pzc}) was determined according to Faria *et al.*²²⁾ The calcium oxide content (%) was calculated from the yield after calcination, using Equation (1)²³⁾.

$$C = 100 \times \frac{M_0 - M_t}{M_0 - M_\infty} \quad (1)$$

where C is the calcium oxide content (%), M_0 is the initial weight of the material (g), M_t is the yield at each temperature (g), and M_∞ is the ideal yield (g).

The elemental distribution on the FC surface before and after phosphate ion adsorption was analyzed using a field-emission electron probe microanalyzer (EPMA; JXA-8530F, JEOL, Tokyo, Japan) operated at an acceleration voltage of

15.0 kV and a beam diameter of 5 μm .

Phosphate Ion Adsorption Experiment All the adsorption experiments were conducted using the batch test method described below. Each FC sample (0.05 g) was added to a 1500-mg/L phosphate solution (50 mL). The mixture was agitated under the following conditions (temperature: 25°C, reaction time: 24 h, agitation speed: 100 rpm) on a water-bath shaker. After shaking, the solution was filtered using a 0.45- μm membrane filter. The phosphorus concentration of the solution was measured using inductively coupled plasma-optical emission spectroscopy (ICP-OES; iCAP-7600, Thermo Fisher Scientific Inc., Massachusetts, USA), and the amount of phosphate ions adsorbed was calculated using Equation (2).

$$Q = \frac{(C_0 - C_e)V}{W} \times \frac{M_{\text{PO}_4}}{M_p} \quad (2)$$

where Q is the amount of phosphate ions adsorbed (mg/g), C_0 is the initial phosphate ion concentration (mg/L), C_e is the equilibrium phosphate ion concentration (mg/L), V is the solvent volume (L), W is the weight of each adsorbent (g), M_p is the molecular weight of phosphorus, and M_{PO_4} is the molecular weight of the phosphate ions. To evaluate the effect of solution pH on the adsorption capacity, the pH of the phosphate solution was adjusted to 3, 5, 7, 9, and 11 using HCl or NaOH. In addition, to evaluate the effect of temperature on the adsorption capacity, the agitation temperature was set to 7, 25, and 45°C.

Calcium Elution Experiment Each FC (0.05 g) was added to 50 mL of pure water and shaken for 24 h at 100 rpm and 25°C, and then the solution was filtered using a 0.45- μm membrane filter. The amount of calcium ions released was calculated based on the calcium ion concentration in the solution, which was measured using ICP-OES.

RESULTS AND DISCUSSION

Physicochemical Properties of Each FC before Phosphate Adsorption

Thermal Decomposition Behavior

Figure 1 shows the TG-DTA profile of each FC before phosphate adsorption. The thermogravimetric value significantly decreased, and the DTA curve showed an endothermic peak at around 800°C. By combining the TG and DTA curves, a substance's pyrolysis reaction could be predicted.^{24, 25)} The peak pattern observed in this study indicated that calcium carbonate (the main component of FC) was converted to calci-

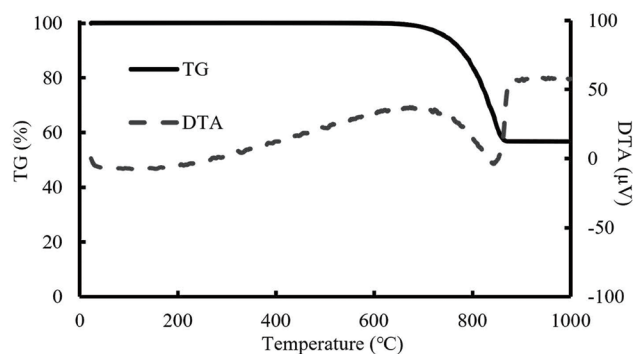


Fig. 1. Thermal Analysis of FC under an Air Atmosphere

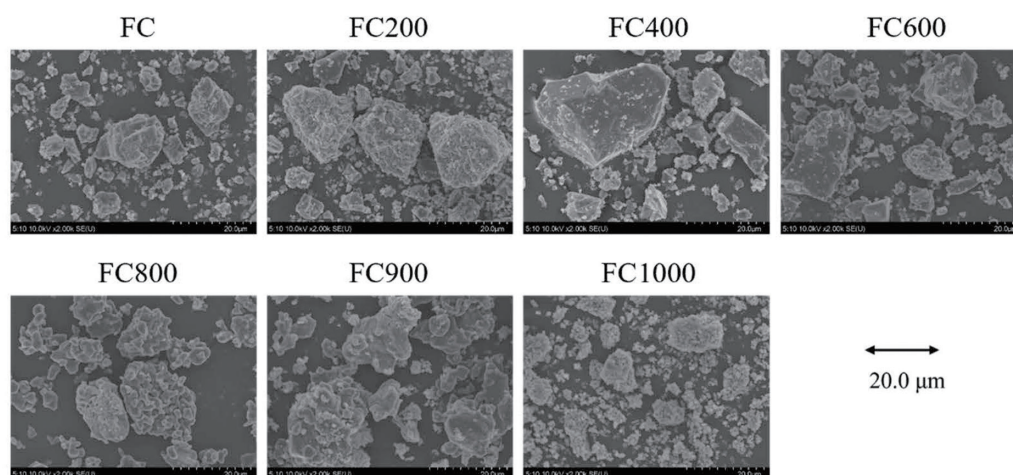


Fig. 2. SEM Images of Each FC before Adsorption

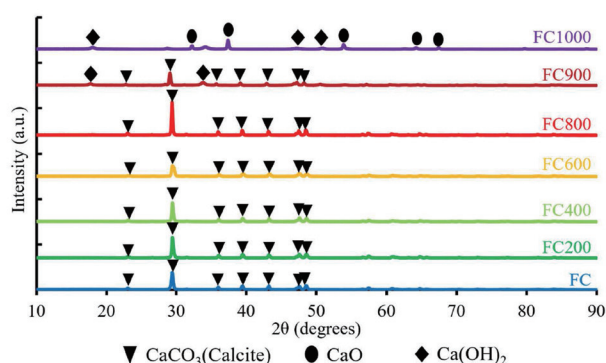


Fig. 3. XRD Patterns of Each FC before Adsorption

um oxide by thermal decomposition at around 800°C. Under atmospheric conditions, the pyrolysis temperature increased due to the influence of the partial pressure of CO_2 .²⁶⁾ In contrast, TG-DTA analysis performed using a small sample size was only slightly influenced by the CO_2 partial pressure and helped identify the optimal pyrolysis temperature.

Changes in Structure and Composition Influenced by Calcination Temperature

The morphology and crystalline structure of FC underwent significant changes depending on the calcination temperature. Figure 2 shows SEM images of each FC before phosphate adsorption. FC, FC200, FC400, and FC600 exhibit smooth surface structures. It has been reported that calcite obtained through the phase transition of aragonite exhibits morphological characteristics distinct from those found in naturally available calcite.²⁷⁾ A distinct crystalline shape was observed in the calcium carbonate reagents, whereas this was not observed in FC, suggesting that trace elements or growth conditions may influence the crystalline morphology. FC900 and FC1000 had many irregularities and structures resembling particle aggregation. FC800 had both structures, suggesting that calcination above 800°C affected the FC structure. Moreover, it has been reported that calcined corals exhibit a relatively rough surface and irregular shape, with some corals bonded together to form large agglomerates.²⁸⁾ This pattern was observed in FC800–

Table 1. Physicochemical Properties of Each FC

Adsorbents	Specific surface area (m^2/g)	CaO (%)	pH_{pzc}
FC	0.77	0.00	9.77
FC200	0.47	0.00	9.60
FC400	0.42	0.00	9.27
FC600	0.37	0.65	9.87
FC800	0.17	4.25	11.14
FC900	2.73	40.40	N/A
FC1000	6.14	97.18	N/A

N/A: not applicable

FC1000, and it is thought that changes in the main components, as described later, were involved. Figure 3 shows the XRD pattern of each FC before adsorption. The peak pattern of calcium carbonate (calcite) was observed in FC, FC200, FC400, FC600, and FC800; both calcium carbonate and calcium hydroxide in FC900; and calcium hydroxide and calcium oxide in FC1000. Previous studies have reported that untreated FC exhibits an aragonite structure,²⁹⁾ which was not the case in the FC of the present study. While TG-DTA conditions suggested pyrolysis initiation at around 800°C, under the sample preparation conditions created using a muffle furnace, decarbonation occurred at temperatures above 900°C, leading to pyrolysis of the main components. Regarding the calcium hydroxide observed in FC900 and FC1000, the calcium oxide generated by pyrolysis was thought to have absorbed moisture from the air and transformed into calcium hydroxide.

Physicochemical Properties and the Amount of Calcium Ions Eluted

Table 1 shows the physicochemical properties of each FC. The specific surface area decreased gradually from FC to FC800 but increased rapidly for FC900 and FC1000. Fuji *et al.* reported that, upon heating, atomic diffusion and realignment led to the sintering of the calcium carbonate surface microstructure, decreasing the microporosity and, consequently, the specific surface area.³⁰⁾ The results of our study are consistent

with this previous study. On the other hand, the increase in the specific surface areas of FC900 and FC1000 may be attributed to decarboxylation, which increased the surface irregularities and made the structure more complex. The pH_{pzc} values of FC–FC800 ranged from 9.27 to 11.14, and under conditions where the solution pH was lower than these values, the adsorbent surface was expected to be positively charged. FC900 and FC1000 were mainly composed of calcium oxide, a strong base, which made determining their pH_{pzc} values difficult. The calcium oxide content (%) was calculated from the weights before and after calcination, and the results exhibited a trend similar to that of the amount of calcium ions released. Figure 4 shows the amount of calcium ions released by each FC. The amount of calcium ions released increased with increasing calcination temperature, particularly for FC900 and FC1000, which showed high values of 136.0 and 431.6 mg/g, respectively. These high values are attributable to the relatively high solubility of calcium oxide (approximately 1.2 g/L), which is the main component of FC900 and FC1000, compared to the much lower solubility of calcium carbonate (approximately 0.013 g/L). Therefore, the measured release reflected the calcium oxide content of each sample.

Evaluation of Phosphate Adsorption Performance

Maximum Adsorption Capacity

Figure 5 shows the amounts of saturated phosphate ions adsorbed. The amount of phosphate ions adsorbed by FC1000 showed the highest value. FC200–FC600 showed a decreasing trend with increasing calcining temperature, whereas the

amount adsorbed by FC800–FC1000 showed an increasing trend with increasing calcining temperature. Previous studies have reported that the calcite structure can act as a seed crystal for HAp,³¹⁾ and HAp crystal growth may be associated with the adsorption mechanisms of FC–FC800. The high adsorption capacity of FC1000 can be attributed to it being mainly composed of calcium oxide, which is highly soluble and provides the numerous calcium ions needed for HAp formation. After phosphate ion adsorption by FC–FC900, the pH showed weak acidity, ranging from 6.4 to 6.5, whereas FC1000 exhibited strong basicity (at 11.9). These results suggest that, although FC1000 exhibits excellent adsorption capacity, it renders the solution strongly basic; therefore, FC200 is more suitable for use in aquatic environments.

Adsorption Mechanism Analysis

Figure 6 shows the results of a qualitative analysis of each FC surface before and after the adsorption of phosphate ions. The phosphorus signal intensity increased after adsorption in all FC samples, indicating that phosphate ions were adsorbed onto the FC surfaces. The composition of FCs differed between the surface and core because of prolonged internal recrystallization and replacement (diagenesis) over time.³²⁾ Consequently, highly adsorbed regions may be localized on the FC surface. Figure 7 shows the XRD patterns of each FC after adsorption. A peak pattern corresponding to brushite (calcium hydrogen phosphate) was observed in all the FC samples, and in FC900 and FC1000, additional peaks attributable

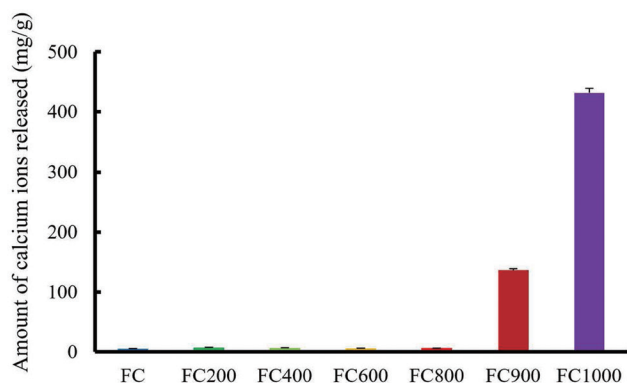


Fig. 4. Amount of Calcium Ions Released

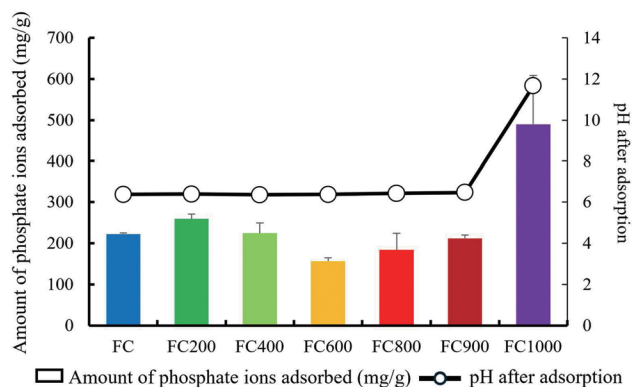


Fig. 5. Amount of Phosphate Ions Adsorbed for each FC

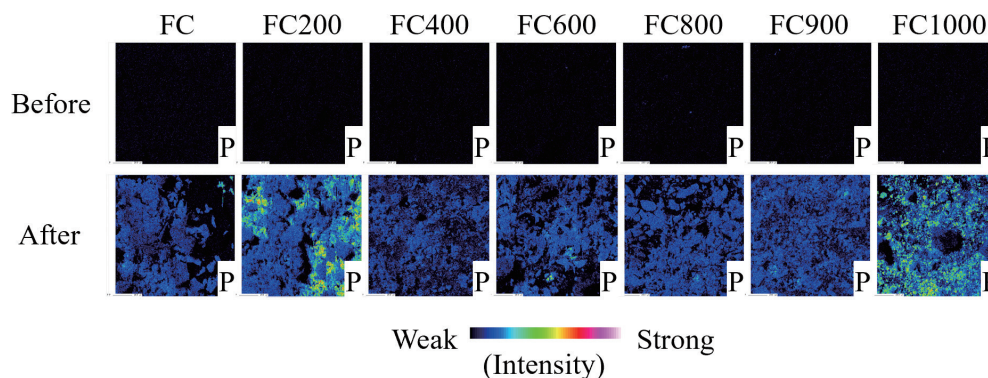


Fig. 6. Qualitative Analysis of Adsorbent Surface before and after Adsorption of Phosphate Ions

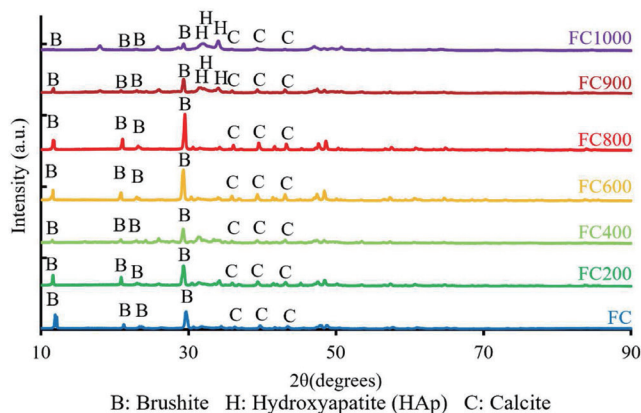


Fig. 7. XRD Patterns of Each FC after Adsorption of Phosphate Ions

to HAp were also observed. Generally, the crystalline phase of calcium phosphate precipitates varied depending on the pH.^{33, 34} Under acidic conditions, HAp formation was inhibited and brushite or octacalcium phosphate was preferentially formed,³⁵ whereas HAp precipitates under strongly basic conditions. The detection of a brushite peak in FC–FC800, which exhibited a weakly acidic pH after adsorption, is consistent with the results of previous studies. On the other hand, after adsorption using FC1000, the solution exhibited strong basicity, which is consistent with the conditions required for HAp formation. These results are consistent with those of previous studies. Additionally, calcite peaks were observed for both FC900 and FC1000. This was thought to have been formed by the carbonation of calcium hydroxide in solution.³⁶

Figure 8 shows the effect of pH on the amount of phosphate ions adsorbed. The amounts of phosphate ions adsorbed by FC–FC800 were higher at acidic conditions (pH 3), and a decreasing trend was observed with an increase in the initial pH. The initial pH values, at which high amounts of phosphate ions were adsorbed, were all below the pH_{pzc} value of each FC sample (9.27–11.14). In other words, for FC–FC800 (with low calcium elution), HAp crystallization did not occur and adsorption onto the surface played a dominant role via electrostatic interactions. Meanwhile, the amount of phosphate ions adsorbed by FC1000 was the highest at an initial pH of 11 and showed a decreasing trend with a decrease in the initial pH. As mentioned previously, HAp precipitation occurred in FC1000, for which the solution exhibited strong basicity after adsorption. The main mechanism was assumed to be the coprecipitation of calcium phosphate by calcium ions and phosphate ions, and the involvement of this mechanism contributed to a large amount of phosphate ions being adsorbed. The results for FC900 indicate that, at a low pH, the surface became positively charged, as observed for FC–FC800, leading to adsorption; however, at a high pH, calcium phosphate formation occurred in a manner similar to that in FC1000, resulting in an apparent reduction in the pH dependence of adsorption.

Figure 9 shows the effect of temperature on the amount of phosphate ions adsorbed. The amount of phosphate ions adsorbed by FC, FC800, and FC900 showed the highest values at 7°C and lowest at 25°C. In contrast, the amount of phosphate ions adsorbed by FC200, FC400, and FC600 increased

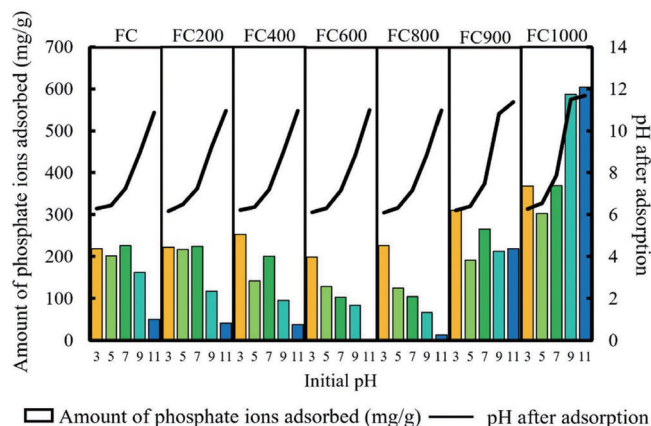


Fig. 8. Effect of pH on the Amount of Phosphate Ions Adsorbed

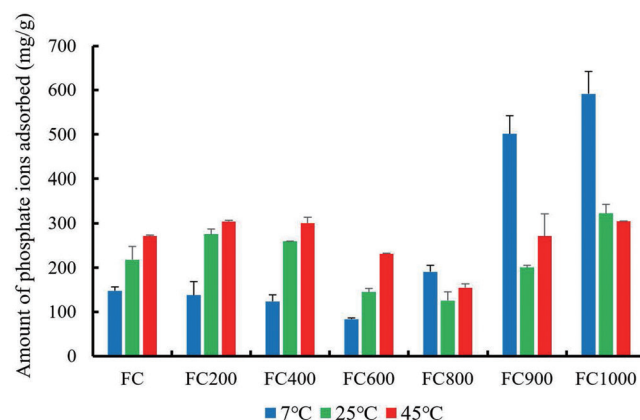


Fig. 9. Effect of Temperature on the Amount of Phosphate Ions Adsorbed

with increasing temperature, reaching a maximum at 45°C. The increase in the adsorption capacity with increasing temperature can be attributed to an increase in the reaction rate and the involvement of chemisorption.³⁷ Therefore, the adsorption behaviors of FC200, FC400, and FC600 suggest the involvement of chemisorption. In addition, the amount of phosphate ions adsorbed by FC1000 showed the highest value at 7°C and decreased with increasing temperature. The high adsorption capacity of FC1000 at low temperatures can be attributed to the enhanced dissolution of calcium oxide, which dissolves exothermically in water. Furthermore, based on the TG-DTA results (Figure 1), FC was assumed to contain calcium oxide when calcined at temperatures above 800°C. Therefore, the greater adsorption observed at 7°C than 25°C for FC900 was also attributed to the enhanced dissolution of calcium oxide, as in the case of FC1000.

Conclusion This study evaluated FC as a phosphorus adsorbent to promote the recycling of phosphorus resources. TG-DTA and XRD showed that the main component, calcium carbonate (calcite), decomposed at 800–900°C to form calcium oxide. FC1000, which was dominated by calcium oxide, exhibited markedly enhanced calcium leaching, indicating that dissolved calcium ions acted as a key source for phosphate removal. Calcite in FC was favorable for phosphate adsorp-

tion and HAP formation. Meanwhile, EPMA confirmed phosphate accumulation on the FC surface, and the adsorption capacities were particularly high for FC200 and FC1000. For FC200, adsorption was attributed mainly to brushite crystal growth from the calcite structure, whereas for FC1000, it proceeded via both HAP formation through the coprecipitation of dissolved calcium and phosphate ions and additional surface adsorption. These findings indicate that FCs are useful materials for phosphate removal and that calcination can be used to tailor their adsorption performance, although further studies are needed to assess their reuse as a fertilizer.

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Data availability The datasets used during the current study are available from corresponding author on reasonable request.

Competing interests The authors declare no competing interests.

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