

Original Article

Effects of pH and Oxygen on Phosphorus Release from Agricultural Drainage Ditch Sediment in Reclaimed Land, Kasaoka Bay, Japan

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ABSTRACT

Agricultural farms are considered to be non-point sources of phosphorus (P) loads to rivers and lakes. Sediment can act as a source or sink of P under different environmental conditions. Inorganic P is the major form, and is a very useful indicator to evaluate the potential release of P in sediment. This study aimed to evaluate the effects of pH and aerobic/anaerobic conditions on P release from sediment by the changes of P fractions before and after incubation. Sediment samples were collected from a drainage ditch of a livestock farm on reclaimed land in Kasaoka Bay, Japan. Experiments were conducted in a 200-mL glass serum bottle with 8 – 10 g fresh sediment and 100 mL of 0.02 M KCl solution, adjusted to pH 4, 7, or 10, and incubated under aerobic or anaerobic conditions. Phosphorus was released under both aerobic and anaerobic conditions until day 5. The amount of P released under anaerobic conditions increased continuously towards day 10 while that under aerobic conditions decreased after day 5 to the end of experiment ($p < 0.05$). Thus, sediment acted as a sink of P under aerobic conditions and as a source of P under anaerobic conditions. Sediment fractionations indicated that loosely sorbed P (loosely-P) and iron-bound P (Fe-P) fractions were the main sources of P released to the overlying water. The amount of P released from sediment at acidic pH was higher than that at neutral or alkaline pH under anaerobic conditions ($p < 0.05$). However, under aerobic conditions, the amount of P release was higher at alkaline pH than that at acidic or neutral pH ($p < 0.05$).

Keywords: aerobic, agricultural sediment, anaerobic, pH, phosphorus release

INTRODUCTION

Phosphorus (P) is one of the most limiting nutrients contributing to eutrophication of both fresh and estuarine surface waters [1,2]. Excessive application of P on agricultural farms as a fertilizer results in eutrophication of downstream lakes and seas. Agricultural farms are considered to be non-point sources of nutrient loads of rivers and lakes, and drainage ditches are the pathways of nutrient loads between farmland and recipient aquatic systems [3–6]. In agricultural ditches, the interactions between sediment and overlaying water involve a number of complex processes, one of which is the release of P from sediment into overlaying water.

Sediment can act as a source or sink of phosphorus depending on environmental conditions, such as pH, redox potential, and temperature. Sedimentary P has the potential to be

released back into the water column [2,7], which may significantly impact water quality. An improved understanding of the mechanism of P release would assist in controlling eutrophication. Inorganic P (IP) in sediment is the major form of P [8], and it can be divided into four fractions: loosely sorbed P (loosely-P), iron-bound P (Fe-P), aluminum-bound P (Al-P), and calcium-bound P (Ca-P). These P fractions are very useful indicators of the potential to release P from sediment. Studies on P-binding forms are also crucial in predicting how the system will react under changing conditions [9,10]. The loosely-P fraction includes P dissolved in the pore water. Sun *et al.* [11] and Wang *et al.* [8] reported that the concentrations of loosely-P were only 1 – 3% and 0.1 – 3.5% of IP in the Haihe River in shallow lake sediments in China, respectively. Iron-bound P represents the redox-sensitive P fraction, which is considered a potentially mobile pool of P [12].

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Oxygen level is one of the most important factors affecting the process of sorption and desorption of P in sediment [2]. Hupfer and Lewandowski [13] reviewed that oxygen is a key factor in controlling P release from sediment because the sorption of oxidized ferric complexes for P is destroyed as oxygen concentration is depleted resulting in P release. Whereas, aerobic sediment increases a high sorption capacity of sediment due to retaining P to a mono-layer of Fe(OOH). Reduction-oxidation reactions play a critical role in the fate of P [14]. Phosphorus fraction forms are sensitive to changes in pH and oxygen levels leading to the release of P in sediment. P-rich sediment in lakes and rivers may cause surface water pollution. In addition, sediment and water in lakes and rivers are sometimes polluted by discharge of agricultural waste. However, researchers pay less attention to drainage ditches. The release of P in lake sediments is well documented, but little information about P release from drainage ditch sediment has been reported. Especially, the contribution of different P fractions in ditch sediments to water is not fully understood yet.

Previous studies focused mainly on the effect of drainage ditch sediment on P retention capacity [5,15]. The release of P from sediment can be estimated by the change of P fractions before and after P release experiments [8]. Various types of P minerals can form in sediments, depending on the physiochemical conditions [16]. These different fractions (forms of P) were attributed to the different sediment characteristics such as diagenesis, and environmental conditions affected the P amounts and forms of P release. For example, Fe-P and Al-P were the main fractions released (80%) into the overlying water from sediments in shallow lakes in China [8]. In addition to P released from sediment of Lake Meiliang and Gonghu, it was released only under anaerobic conditions and released forms were mainly from Fe-P, Ca-P, and Al-P fractions [2]. On the other hand, Jiang *et al.* [17] reported that Ca-P and organic P (OP) in sediment decreased under both aerobic and anaerobic conditions, but Fe-P and Al-P increased under aerobic conditions.

The drainage ditches in Kasaoka may have special characteristics because of its diagenesis (reclaimed farm) and it differs from lake and river sediments under alkaline conditions. To improve the water quality, it is necessary to reduce the sources of nutrients. The best management practices are needed to assess in improving nutrient retention. However, the best management practices are currently applied without sampling and characterization of ditch soils and their geomorphic environment [4]. It must be noted that P release is roughly equivalent to external loads [16]. Knowledge of

the factors influencing P release is needed because P is the limiting factor for eutrophication. We need to develop a better understanding of the P release mechanism in agricultural drainage sediment so that eutrophication downstream is controlled. The aim of this study was to evaluate P release mechanisms under changing pH and aerobic/anaerobic conditions with special attention to P fractions released from sediment before and after incubation.

MATERIALS AND METHODS

Study area

Kasaoka Bay is located in southwestern Okayama Prefecture (34°28'59" N, 133°29'30" E). Agricultural activities on reclaimed land from Kasaoka Bay with an area of 1,191 ha were established in 1990. This area has the main component of water transfer networks, which redistributes water within farmlands and links fields with receiving aquatic systems. The area in the northwest part is divided into three sectors: 176 ha of livestock, 99 ha of crop fields, and 132 ha of grasslands. The annual precipitation and temperature of the study area from 2000 to 2014 is 1,008 mm and 15.4°C, respectively, which was obtained from the Automated Meteorological Data Acquisition System (AMeDAS). (<http://www.jma.go.jp/en/amedas/>).

Sample collection

The ditch is approximately 1,850 m in length, 3 m in width, and 0.35 m of water level. Sediment samples were collected from the depth of 0 – 15 cm on 21 April 2014 in a drainage ditch that receives effluent with high concentrations of nutrients mostly from livestock farming. Our surveys on water quality since 2008 have demonstrated that the ditch had been contaminated with nitrogen and phosphorus throughout the years. Water and sediment samples were taken in triplicate from the central area at the end of ditch, where higher concentrations of nitrogen and phosphorus were found from previous surveys [18], to develop the understanding for P release mechanisms. Sediment samples were obtained by hand using a core sampler of 5.5 cm diameter. All samples were transported to the laboratory in a cooler box within 3 h.

Phosphorus release experiment

Phosphorus fractions are very sensitive to the change of environmental conditions, of which pH and oxygen levels are considered to be the major factors affecting P release mechanisms in sediment. The pH levels of solutions were adjusted to represent acidic, neutral, and alkaline conditions.

Table 1 Physical and chemical characteristics of drainage ditch sediment and water.

Sediment and water parameters		Values
Sediment	TC [%]	4.2 ± 0.1
	TN [%]	0.33 ± 0.1
	TP [mg/kg]	489.3 ± 0.1
	pH	8.0 ± 0.1
	EC [dS/m]	2.3 ± 0.5
Particle sizes	Clay [%]	3 ± 1.4
	Silt [%]	68 ± 4.1
	Sand (%)	29 ± 2.7
Water	Soluble reactive P [mg/L]	1.4 ± 0.0
	TP [mg/L]	1.8 ± 0.0
	EC [dS/m]	3.86
	pH	8.0

TC: total carbon, TN: total nitrogen, TP: total phosphorus, EC: electrical conductivity.

In addition, incubation temperature was also selected to stimulate the summer temperature because of high temperature promotes P release from sediment [19]. The following experiments were immediately performed in a 200-mL glass serum bottle (5.5 cm in diameter). Eight to 10 grams of homogenized fresh sediment (5–7 g dry equivalent) was loaded into the bottle with 0.02 M KCl solution, adjusted to pH 4, 7, or 10 by HCl or NaOH, and incubated under aerobic or anaerobic conditions. The electrical conductivity (EC) of the drainage water in the field ranged from 2.87 to 4.15 dS/m in 2014. Ionic strength affects P sorption/desorption behaviors. Potassium chloride was added to simulate the ionic strength in the field. For anaerobic treatments, the bottles were capped with a butyl stopper and the water column was purged with N₂ gas until saturation. All bottles were wrapped with aluminum foil and incubated at room temperature (25 ± 1°C) in a dark place to prevent algae development. Sediment and overlying water were sampled on days 1, 2, 3, 4, 5, and 10.

Water samples were filtered through a 0.2 µm filters (DISMIC-25AS, ADVANTEC, Tokyo, Japan) to analyze the concentration of soluble reactive P (SRP). Unfiltered water sample was digested with K₂S₂O₈ and product (PO₄-P) was analyzed for total P (TP). Phosphorus fractions were extracted from sediment samples before and after incubation using the sequential extraction scheme of Rydin [12].

The extraction scheme includes five fractionations of P in sediment. Loosely-P and Fe-P were extracted with 1 M NH₄Cl solution (at pH 7 for 2 h) and with 0.11 M NaHCO₃/Na₂S₂O₄ for 1 h (pH 8), respectively. Subsequently, extraction of the residue was continued with 0.1 M NaOH for 16 h in a shaker, and the extracted solution included two fractions: Al-P and

organic P (Org-P). The Al-P fraction was measured directly from the extracted solution and other organic phosphates, and the difference between TP digested by K₂S₂O₈ and Al-P, is Org-P. Calcium-bound P was obtained with 0.5 M HCl for 16 h. Extracted suspensions were centrifuged 3,000 rotations per minute (rpm), and the filtrates were filtered through a 0.2 µm membrane filter. Phosphorus of all samples was determined by spectrophotometry using a continuous flow autoanalyzer (QuAAtro 2-HR, Bltec, Tokyo, Japan). The TC and TN were determined with C:N Coder (MT-700, Yanaco, Kyoto, Japan). Approximately 0.5-g air-dried sediment was digested with sulfuric acid and hydrogen peroxide at 300°C for the analysis of TP [20].

The amounts of P released into the overlying water under different pH and aerobic/anaerobic conditions were analyzed by ANOVA (multi-comparisons Tukey-Kramer HSD post-hoc test). The two-tailed *t*-test was used to evaluate the significant differences between the amounts of P fractions released under aerobic and anaerobic conditions. Statistical comparison tests were conducted at a level of significance of *p* = 0.05.

RESULTS AND DISCUSSION

Sediment and water characteristics of the drainage ditch

Table 1 summarizes the sediment and water characteristics of the drainage ditch. The silt fraction was dominant, accounting for more than two thirds of the sediment particle-size distribution. Kröger and Moore [6] indicated that a high percentage of the silt component was a result of the sediment

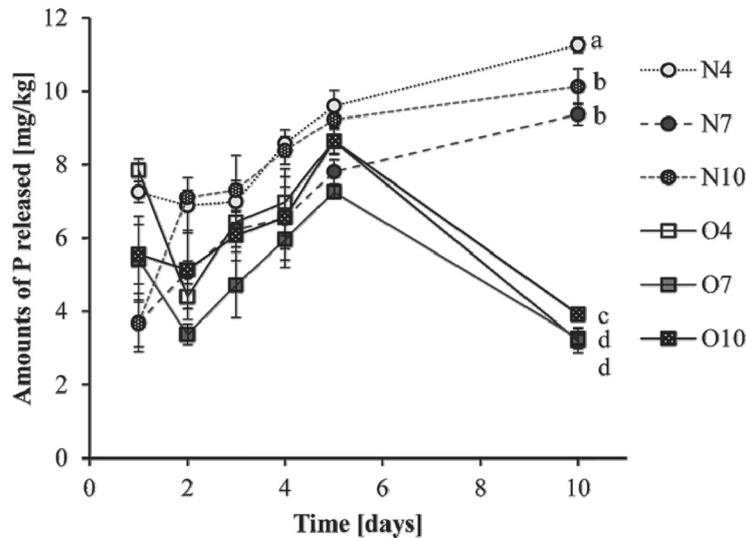


Fig. 1 Amounts of P released under different pH and oxygen conditions.

O4, O7, and O10: Aerobic condition at pH 4, 7, and 10, respectively.

N4, N7, and N10: Anaerobic condition at pH 4, 7, and 10, respectively.

^{a-d}Data with the same letters were not significantly different within each treatment under aerobic or anaerobic conditions at $p > 0.05$.

runoff from agricultural areas. The concentration of SRP agricultural drainage water in the Mississippi Alluvial Valley was 0.08 – 1.30 mg/L [6], in the Manokin River watershed was 0.37 mg/L [21], in New Zealand was from 0.006 – 0.019 mg/L [22], and in the UK was from 0.2 – 0.7 mg/L [23], lower than the value of the present study. This demonstrated that livestock farming in Kasaoka supplied a larger quantity of P to the drainage ditch.

Amounts of P released into overlying water

Figure 1 shows the amount of $\text{PO}_4\text{-P}$ released, which was calculated from the $\text{PO}_4\text{-P}$ concentration in water during the experiment. Phosphorus was released under both aerobic and anaerobic conditions, but the magnitudes were different. On the first 5 days, all treatments showed a similar trend in the release of P from sediment. This indicates that P can be released from sediment, regardless of pH (4, 7, or 10) and aerobic/anaerobic conditions. In general, the amount of P released during the 10-day incubation was greater in acidic pH than that of alkaline or neutral pH under anaerobic conditions ($p < 0.05$). In contrast, the amounts of P released were significantly higher at alkaline pH than that at acidic or neutral pH under aerobic conditions ($p < 0.05$).

Under anaerobic conditions, the concentrations of P increased remarkably over the experiment. The amounts of P released at pH 4, 7, and 10 were 11.3, 9.4, and 10.1 mg/kg on

day 10, respectively. There was no significant difference in the amounts of P released between pH 7 and pH 10 ($p > 0.05$). The release was quick on the first day of incubation (7.3 mgP/kg) at pH 4, which was higher than that at pH 7 (3.7 mgP/kg) or pH 10 (3.7 mgP/kg). However, the released amounts of P at pH 7 and pH 10 were greater than that of pH 4 from day 1 to day 5. It means that the initial acidic pH affected the solubility of P on the first day of incubation, and then pH increased/decreased towards the sediment pH after 5 days of incubation (**Fig. 2B**). Lower amount of P released at pH 4 from day 1 to day 5 was attributed to higher concentration of P in the water column, which probably regulated P released from sediment. The net amounts of P released under anaerobic conditions from day 5 to day 10 were 1.7, 1.6 and 0.9 mg/kg in acidic, neutral and alkaline treatments, respectively, much lower than those from day 1 to day 5.

In contrast, under aerobic conditions, the release of P gradually increased and reached to 8.8, 7.3, and 8.7 mg/kg on day 5, then decreased to 3.2, 3.3, and 3.9 mg/kg by the end of the experiment at pH 4, 7, and 10, respectively. The amount of P released from sediment to the overlying water was not significantly different between pH 4 and pH 7 treatments ($p > 0.05$). Sediment acted as a sink of P after day 5 due to precipitation of Fe with released P, although P was released from sediment from day 1 to day 5. The results agreed with Gao [24], who found that higher amount of P was released from

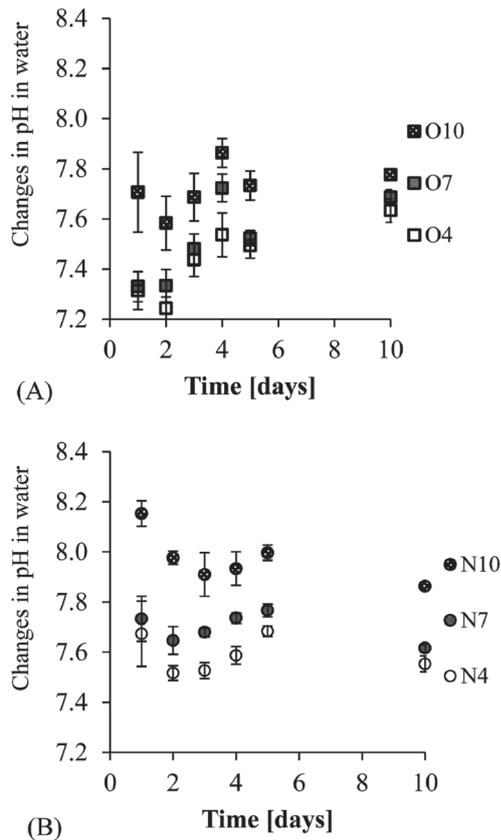


Fig. 2 Change in pH during the experiment. (A): aerobic, (B): anaerobic.

O4, O7, and O10: aerobic condition at pH 4, 7, and 10, respectively.

N4, N7, and N10: anaerobic condition at pH 4, 7, and 10, respectively.

Rongcheng Swan Lake sediments under acidic conditions than that under alkaline or neutral conditions. This study indicates that initial pH plays an important role on P release from sediment. However, the P release showed opposite trends after 5 days of incubation. Probably, P release-course from sediment of the present study can be divided into two stages. The first stage was described as release patterns of P from day 1 to day 5, regardless of pH levels or aerobic/anaerobic conditions. At the second stage, P was slowly released under anaerobic conditions or quick adsorption under aerobic conditions.

The effect of pH on P release was greater at the first stage. Oxygen concentration was important at the second stage. As a result, the effects of pH associated with anaerobic conditions were greater than the effects of pH associated with aerobic conditions on P released from sediment. Similarly, it was confirmed that a low-pH water was more favorable for P

release into water under anaerobic conditions [25]. Because large amounts of P were released under acidic and reductive conditions, it can be concluded that redox reactions play an important role in the regulation of P in sediment. The reduction of Fe^{3+} to Fe^{2+} that results in P release from sediment occurs more strongly under extremely low redox potential and low pH (5.5) conditions [1,26]. On the other hand, lower amounts of P were released under anaerobic conditions at the second stage than at the first stage. Moore *et al.* [16] demonstrated that high concentration of P in the overlying water regulated the release of P from sediment. In addition, the solubility of P precipitation with Al or Ca is controlled by P concentration in water and the solubility of P is lower at neutral pH [27]. Under aerobic conditions, sediment acted as a sink of P after day 5, because Fe is in the form of $\text{Fe}(\text{OOH})$, which has a high capacity to sorb P [7]. Moore and Reddy [26] also reported Fe phosphate precipitation or adsorption of P by Fe oxides or hydroxides because ferric phosphate, such as strengite precipitate, resulting in low P solubility.

The amount of loosely-P released was significantly higher under anaerobic conditions than aerobic conditions in the present study. This would be attributed to the effects of anaerobic conditions on the sorption-desorption equilibrium [2]. Our results suggest that only loosely-P and Fe-P had significant roles in the regulation of P in drainage ditch sediment under both aerobic and anaerobic conditions.

Changes in P concentrations in different sediment fractions during incubation

Tables 2 and 3 illustrate the changes in the amounts of different P fractions in sediments before and after 10 days of incubation under aerobic and anaerobic conditions. The initial P concentrations in different fractions of sediment were in the order: Ca-P > Al-P > Fe-P > loosely-P. The distribution pattern was similar to that in the Haihe River, China [11] and in Volvi and Koronia Lakes, Greece [28].

The loosely-P fraction contributed 10% of IP in sediment. The concentration of Fe-P in sediment was 17.5 mg/kg (11% of IP), higher than the previous report [24], which documented only 3.6 – 10.1 mg/kg of Fe-P in Haihe River sediments, much lower than that in shallow lakes in China (48 – 112 mg/kg) [8]. The Ca-P fraction occupied more than 50% of total IP in sediment. Kaiserli *et al.* [28] found that the concentrations of Ca-P in Volvi and Koronia lake sediments ranged from 59 – 74% of IP. According to previous works [11,24,29], Ca-P was a dominant fraction in carbonate-rich lake sediments. The high concentration of Ca-P in sediment was due to the calcareous recharge [11]. Drainage water in

Table 2 P fractions in sediment before and after 10-day incubation [mg/kg].

P fractions	Before incubation	After aerobic incubation		After anaerobic incubation			
		pH 4	pH 7	pH 10	pH 4	pH 7	pH 10
loosely-P	16.1 ± 0.3	13.6 ± 0.2 ^a	12.3 ± 1.6 ^a	13.7 ± 0.9 ^a	7.2 ± 0.5 ^b	7.4 ± 1.5 ^b	7.7 ± 0.4 ^b
Fe-P	17.5 ± 1.7	16.6 ± 1.3 ^a	18.0 ± 0.1 ^a	17.7 ± 1.4 ^a	14.6 ± 0.1 ^b	14.9 ± 1.1 ^b	15.0 ± 1.3 ^b
Al-P	25.5 ± 1.0	26.0 ± 5.2 ^a	27.5 ± 2.3 ^a	26.3 ± 2.8 ^a	25.9 ± 3.7 ^a	28.2 ± 3.8 ^a	27.7 ± 2.9 ^a
Ca-P	99.2 ± 4.2	101.9 ± 9.0 ^a	98.1 ± 6.2 ^a	100.6 ± 10.3 ^a	98.8 ± 8.9 ^a	93.4 ± 17.5 ^a	95.6 ± 4.6 ^a
Org-P	22.7 ± 3.3	24.5 ± 3.5 ^a	25.2 ± 5.5 ^a	22.4 ± 3.3 ^a	24.6 ± 0.7 ^a	24.4 ± 3.2 ^a	23.1 ± 3.9 ^a

The amounts of P released are expressed as mean ± standard deviation. Data with the same letters ^{a, b} are not significantly different between aerobic and anaerobic conditions at the same pH ($p > 0.05$; *t*-test for comparison).

Table 3 Loosely-P and Fe-P fractions in sediment and the total amount of P released from sediment under anaerobic conditions after 10-day incubation.

	pH 4	pH 7	pH 10
Loosely-P [mg/kg]	8.9 ± 0.5	8.7 ± 1.5	8.4 ± 0.4
Fe-P [mg/kg]	2.9 ± 0.1	2.6 ± 1.1	2.5 ± 1.2
Total released amount [mg/kg]	11.3 ± 0.2	9.4 ± 0.3	10.1 ± 0.5

the present study site is not only receiving discharge from livestock farms but also from the sea-water through the intake canal for dilution, and it may have a high concentration of calcium carbonate.

It seems that only loosely-P fraction was released from sediment into the overlying water under aerobic conditions. However, **Table 3** indicates that both loosely-P and Fe-P fractions were the main components contributing to the increase in the concentrations of P in the overlying water under anaerobic incubation. The contribution of loosely-P was 3 times higher than that of Fe-P. Higher P release only occurred in anaerobic conditions due to reduction of Fe³⁺ to Fe²⁺, resulting in P release from sediment into the overlying water. In addition, pH is a factor controlling the solubility of P: as pH decreased the solubility of P increased [7].

The effect of pH on P release was also reflected through the P speciation in combination with metal oxides or hydroxides and was exchangeable with OH⁻. These are the reasons why the amounts of P release were higher at acidic and alkaline conditions than that at neutral conditions. On the other hand, Al-P and Ca-P fractions in sediment remained unchanged after incubation. The Ca-P fraction, which represents the P fraction sensitive to low pH, seemed to be stable in sediment [8,28,30], although it was released in sediment when maintained at extremely low pH 3 [24]. Moreover, Ca-P was considered to be the immobile form without biological activities [1,7]. Wang *et al.* [2] concluded that Ca-P and OP of sediment from Lake Meiliang did not change after incubation under anaerobic conditions. Accordingly, anaerobic

conditions associated with low pH enhanced P release during the incubation.

Changes in pH during incubation

Figure 2 shows the buffer capacity of sediment and reactions under oxidized and reduced conditions in controlling pH in water column. Although pH values of added solutions were adjusted at 4, 7, or 10 before the experiment, they tended to either increase or decrease towards the original pH level of sediment on the first day of incubation. However, changes in pH levels during incubation under anaerobic conditions were higher than those of aerobic conditions (**Figs. 2A** and **2B**) as a result of denitrification [31] because of the reduction of nitrate to gaseous nitrogen with an organic substrate as an electron donor. Consequently, carbon dioxide and hydroxide (OH⁻) were produced, which may form bicarbonate (HCO₃⁻) and carbonate (CO₃²⁻) [32].

Wang *et al.* [2] also suggested that the rapid increase in pH within one day in the overlying water occurred for the sediments at different oxygen supply levels. According to Jiang *et al.* [17], the changes of pH during incubation were due to the photosynthetic and denitrification processes. We incubated samples in the dark so that algae did not develop. On the other hand, the pH of water was controlled by sediment pH because of the soil buffering function. The P release under different pH indicated that the initial pH affected P release from sediment (**Fig. 1**).

CONCLUSIONS

The P release experiments were conducted using agricultural drainage ditch sediments from Kasaoka Bay reclaimed land at three pH values and under aerobic/anaerobic conditions. Results showed that the amounts of P released varied under different pH and O₂ conditions. The P released was significantly higher under anaerobic conditions than those under aerobic conditions. The amount of P released from sediment at acidic pH was higher than that at alkaline or neutral pH or was higher at alkaline pH than acidic or neutral pH. Loosely-P and Fe-P fractions were the main contributors to P in the overlying water. High amounts of P released to water under anaerobic conditions were associated with low pH because of the reduction of Fe oxy-hydroxides. The results suggest that drainage ditches should be maintained in the presence of oxygen to diminish the concentration of P in surface water.

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