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Recycling of Phosphorus Resources in Agricultural Areas Using Woody Biomass and Biogenic Iron Oxides

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1. Introduction

Phosphorus (P) is an essential element in plant nutrients, because many biochemical processes such as photosynthesis, respiration, and energy transfer depend on inorganic P or its organic derivatives. However, P is difficult for plants to obtain from the rhizosphere and P deficiency is one of the major limitations on crop production. This is because soluble P in soil, the primary P source for plants, is extremely low concentration (Condon et al., 2005) and significant portions of P in the soil are various organic complexes and unavailable (Raghothama, 2005). On a worldwide scale, land covering 5.7 billion hectares is estimated to be deficient in P for optimal crop production (Batjes, 1997). Since the soluble P in the soil is easily taken up by plants and microorganisms, continuous application of P fertilizer is necessary for crop production.

The global demand for P has increased 10-fold since the beginning of the 20th century (Cordell et al. 2009) and approximately 80% of the demand is for agricultural fertilizers (Steen, 1998). Thus, more P will be required as the world's population increases. However, there is concern that world P resources will be depleted in the next 50–100 years, because the reserves of high-grade phosphate rock are limited (Runge-Metzger, 1995; Steen, 1998; Smil, 2000; Stewart et al., 2005). Therefore, the recovery of P is essential for sustaining food production.

Figure 1 shows a conceptual illustration of the global P cycle, which is completed by P flux from the ocean to the land, and is intimately linked to global ocean circulation. The P derived from weathering or fertilizer application on the land is washed down in rivers and enters the ocean food chain. In deep ocean water (about 2,000–3,000 m in depth), the P concentration is considerably higher than that at the surface because dead fish and plankton fall on the ocean floor. However, the P-rich water is too deep for humans to exploit. In the deep ocean, the water flows from the Atlantic Ocean to the Pacific Ocean via the Antarctic and the Indian Ocean, while the surface water flows in the opposite direction. This movement is very slow; about 2000 years is required to complete this circulation. In some areas of the Pacific Ocean, the flow rises from the bottom to the surface, but this is rare phenomenon. Because the occurrence of this rising flow depends on a complex combination of sea currents, winds, and geographical features. Consequently, these selected areas are abundant in plankton and fish. However, the P flux from ocean to land occurs only via

fishery and seabirds' droppings (guano), unlike nitrogen that can be released into the atmosphere via denitrification. In addition, the seabed is gradually transformed into land by the geological movement of the Earth's crust, but this occurs on a much longer time scale than do human activities. Therefore, the global P cycle is extremely limited.

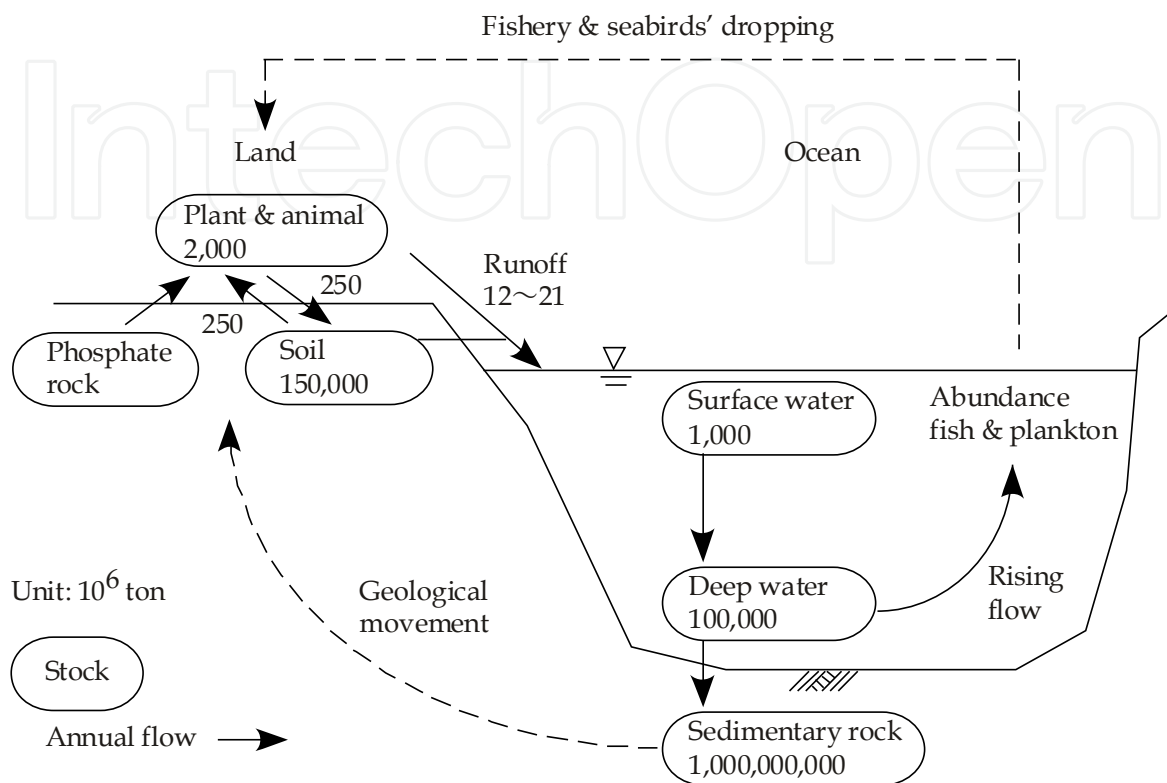


Fig. 1. Conceptual illustration of the global P cycle (data from Sumi, 1989)

Despite the limited nature of the P cycle, repeated applications of fertilizers and organic matter builds up nutrients in the soil. Strong relationships between the level of P monitored by soil tests and the amount of P lost in runoff have been reported (Pote et al., 1996; Sharpley, 1995). Thus, excessive application of P fertilizers contributes to eutrophication, which is sometimes responsible for the lack of clean water resources. From this viewpoint, the recovery of P is also essential.

The behavior of P in nature has been affected by iron (Fe) oxides since ancient time (Bjerrum & Canfield, 2002). In natural water bodies such as canals, swamps, and ponds with low oxygen groundwater seeps and circumneutral conditions, the accumulation of soft, reddish-brown sediment is often observed (Fig. 2). The essential compounds in this sediment are biogenic Fe oxides produced by microaerobic Fe-oxidizing bacteria (Emerson et al., 1999; Emerson & Weiss, 2004; James & Ferris, 2004) and this ferric substance in the sediment can adsorb P in a similar manner to abiotic P adsorbents of ferric compounds (Boujelben et al., 2008; Persson et al., 1996; Seida & Nakano, 2002; Zeng et al., 2004). Therefore, biogenic Fe oxides in nature are considered as one of the P resources. However, they have not yet been recognized as such, although they have been used for ferrous Fe removal in water treatment facilities (Pacini et al., 2005; Katsoyiannis & Zouboulis, 2004; Søgaard et al., 2001). This is because biogenic Fe oxides in natural water bodies are easily dispersed by water turbulence. In addition, it is difficult to collect only the Fe oxides as a P resource, because they usually

accumulate only a few centimetres, and anaerobic and malodorous mud exists underneath (see Fig. 3). Moreover, the mud deposits that have existed for a long time may accumulate harmful substances such as heavy metals.



Fig. 2. Accumulation of reddish-brown soft sediment in an agricultural canal

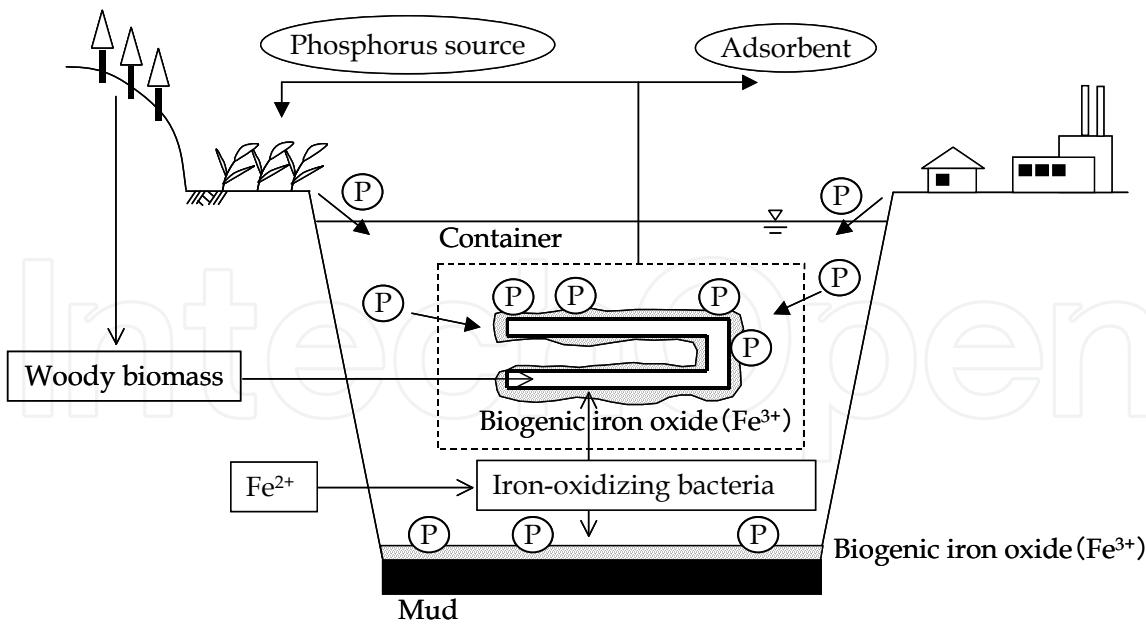


Fig. 3. Conceptual illustration of P recovery from natural water bodies using Fe-oxidizing bacteria and woody biomass.

A new method for the recovery of P from natural water bodies using Fe-oxidizing bacteria and woody biomass as a carrier has been proposed (Fig. 3). A woody carrier is immersed in

water in which Fe-oxidizing bacteria are abundant and then removed several weeks later. In this chapter, this method was tested in an agricultural area, dominated by rice paddy fields, located in the eastern part of Shimane Prefecture, Japan. As the woody carrier, sawdust from the Japanese cedar and Japanese cypress were used. Since the accumulation of biogenic Fe oxides was observed throughout the year at several locations, the water quality at these points was monitored. In addition, heavy metals on the immersed carrier were also measured, because biogenic Fe oxides have the potential to also adsorb heavy metals such as arsenic (As), cadmium (Cd), chromium (Cr), mercury (Hg), lead (Pb), zinc (Zn), and nickel (Ni).

2. Material and methods

2.1 Water quality monitoring

Samples for water quality monitoring were collected at eight points on agricultural drainage canals (Fig. 4) on December 13, 2008. These points were located in the downstream area of the Hii River, Japan, at approximately 35° 24' N and 132° 50' E. The pH and oxidation-reduction potential (ORP) were monitored with a portable analyzer (Kasahara Chemical Instruments, KP-5Z). The Fe, P, and nitrogen (N) concentrations were analyzed in accordance with Japanese Industrial Standard (JIS) K 0102 (Namiki, 2003): total Fe (T-Fe) and dissolved Fe (D-Fe) were measured by the 1,10-phenanthroline method; total phosphorus (T-P) was measured by the ascorbic acid reduction molybdenum blue method after potassium peroxodisulfate decomposition; phosphate phosphorus (PO₄-P) was measured by the ascorbic acid reduction molybdenum blue method; total nitrogen (T-N) was measured by UV absorption spectroscopy after alkaline potassium peroxodisulfate decomposition; ammonium nitrogen (NH₄-N) was measured by the indophenol blue method; nitrate nitrogen (NO₃-N) was measured by ion chromatography (Shimadzu HIC-6A). The total organic carbon (TOC) concentration was measured by Shimadzu TOC-Vcsn system and suspended solids (SS) were measured by gravimetric analysis using glass-fiber filters (pore size = 0.45 μm; Advantec GS25).

2.2 Biomass carrier

Although the precise mechanism of Fe oxidation-deposition by Fe-oxidizing bacteria is not sufficiently understood (Pacini et al. 2005) and some of the species are characterized as autotrophic (Hallbeck & Pedersen, 1991; Imai, 1984), a substantial accumulation of biogenic Fe oxides was found on the surface of submerged aquatic plants in an agricultural drainage canal (Fig. 5). On the basis of this finding and some trial-and-error experiments, woody biomass (conifer heartwood) was used as the carrier for collecting biogenic Fe oxides. In particular, sawdust (particle size: 0.2–2 mm) of the Japanese cedar (*Cryptomeria japonica*) and the Japanese cypress (*Chamaecyparis obtusa*) were used, both of which are typical conifers found in Japan. The heartwood of the conifer contributes very little to secondary water pollution during the immersion test period, because it mainly consists of carbon, hydrogen, and oxygen and contains extremely small amounts of N and P (Jodai & Samejima, 1993). In addition, it contains a large amount of lignin, flavonoids, and phenols, which provide resistance to wood-decomposing fungi (Jodai & Samejima, 1993). Moreover, approximately 97% of the wood tissue of conifer heartwoods consists of tracheids, which are hollow elongated cells (Furuno & Watanabe, 1994). Thus, the sawdust is expected to have a large specific surface area.

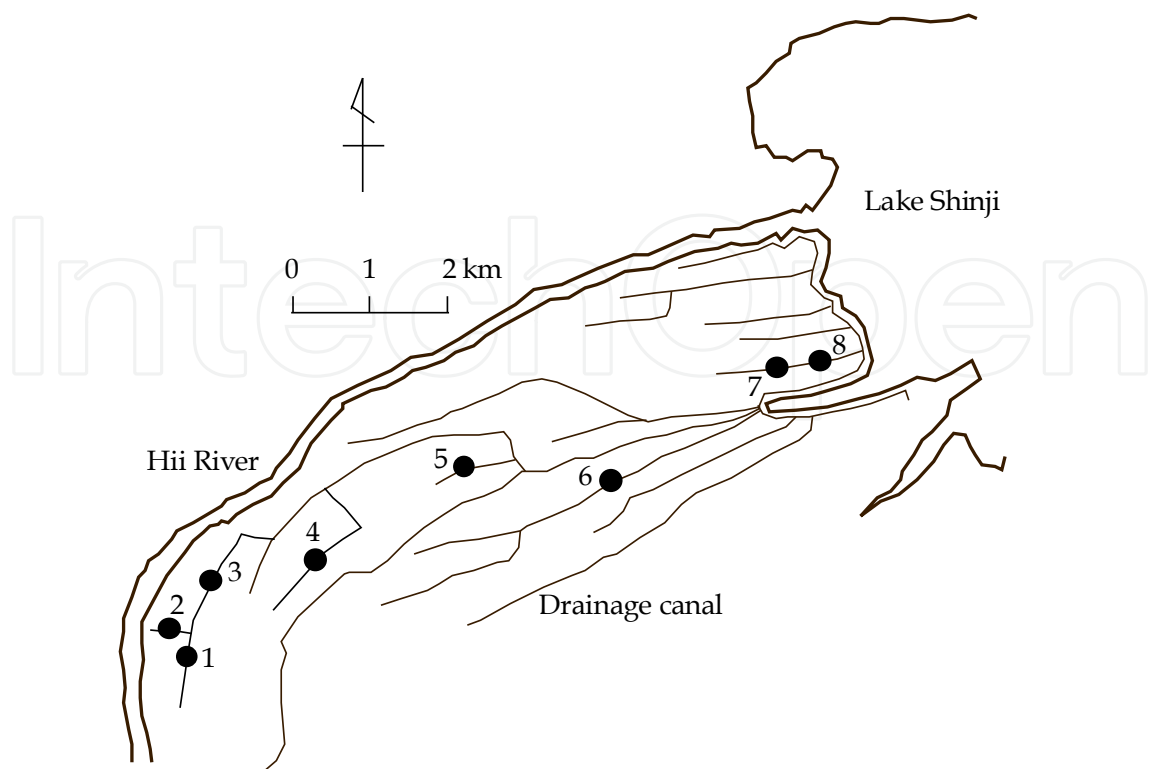


Fig. 4. Map of study site



Fig. 5. Accumulation of biogenic Fe oxides on the surface of submerged aquatic plant



Fig. 6. Sheathed bacteria, *Leptothrix* spp.

2.3 Immersion test

The immersion test was conducted in an agricultural drainage canal (at point 1 in Fig. 4) where reddish-brown sediment accumulated, and sheathed bacteria (*Leptothrix* spp.) were found to be abundant (Fig. 6). The test was performed during the irrigation period for paddy fields (from May to September 2009) and the non-irrigation period (from October 2009 to April 2010), because the canal is mainly fed by drainage water from paddy fields via surface outlets and underdrains, and the water quality is affected by the paddy field irrigation.

In this test, the woody carrier was placed in a container of non-woven bag and lowered to the bottom of the canal. The carrier in the container was removed from the water after immersion for 4 weeks. The Fe collected on the immersed carrier was analyzed by the 1,10-phenanthroline method (Stucki & Anderson, 1981), and the P adsorbed on the Fe oxides was analyzed by the Bray-2 method (Byrnside & Sturgis, 1958). The Bray-2 P is a portion of the soil P and is one of the indexes of available P for plant uptake. In this study, the adsorbed P is expressed as g/kg instead of the conventional expression of Bray-2 P (mg P₂O₅/100 g dry material). In addition, the water samples were collected at weekly intervals and the water quality of D-Fe and PO₄-P was analyzed by the above-mentioned methods.

2.4 Elemental analysis

Elemental analysis of the immersed carrier was carried out by X-ray fluorescence spectrometry system (Shimadzu, EDX-720) at a voltage of 50 kV and a current of 1 mA.

3. Results and discussion

3.1 Water quality in the canals

Table 1 presents the water quality at eight points on the agricultural canals. At all points, the D-Fe concentration was much lower than the T-Fe concentration, and the same relationship was found between the PO₄-P concentration and T-P concentration. Therefore, most of the Fe and P in the water were associated with particulate matter. The average concentrations of

T-N and TOC were 2.864 and 2.180 mg/L, respectively, and the NO₂-N concentration was much lower than the T-N concentration.

Site	pH	ORP (V)	T-Fe (mg/L)	D-Fe (mg/L)	T-P (mg/L)	PO ₄ -P (mg/L)	T-N (mg/L)	NH ₄ -N (mg/L)	NO ₂ -N (mg/L)	NO ₃ -N (mg/L)	TOC (mg/L)
1	6.7	0.02	8.474	0.109	0.180	0.020	3.229	0.653	0.016	2.533	2.483
2	6.8	0.025	4.742	0.235	0.145	0.012	2.585	0.494	0.013	1.840	1.476
3	7.1	-0.026	13.222	0.600	0.319	0.022	2.627	1.293	0.003	1.080	1.833
4	6.9	0.049	12.612	0.085	0.226	0.022	1.972	1.568	0.002	0.169	2.257
5	6.7	-0.017	7.999	0.201	0.261	0.023	2.118	0.968	0.006	1.030	1.421
6	6.8	-0.017	14.783	0.061	0.529	0.033	2.330	1.324	0.013	0.630	2.077
7	6.8	-0.029	16.920	0.085	0.244	0.012	1.738	1.283	0.003	0.000	2.757
8	6.9	-0.015	12.171	0.071	0.180	0.002	6.309	1.114	0.036	4.470	3.138
Mean	6.8	-0.001	11.365	0.181	0.261	0.018	2.864	1.087	0.012	1.469	2.180

Table 1. Water quality at eight points on the agricultural canals

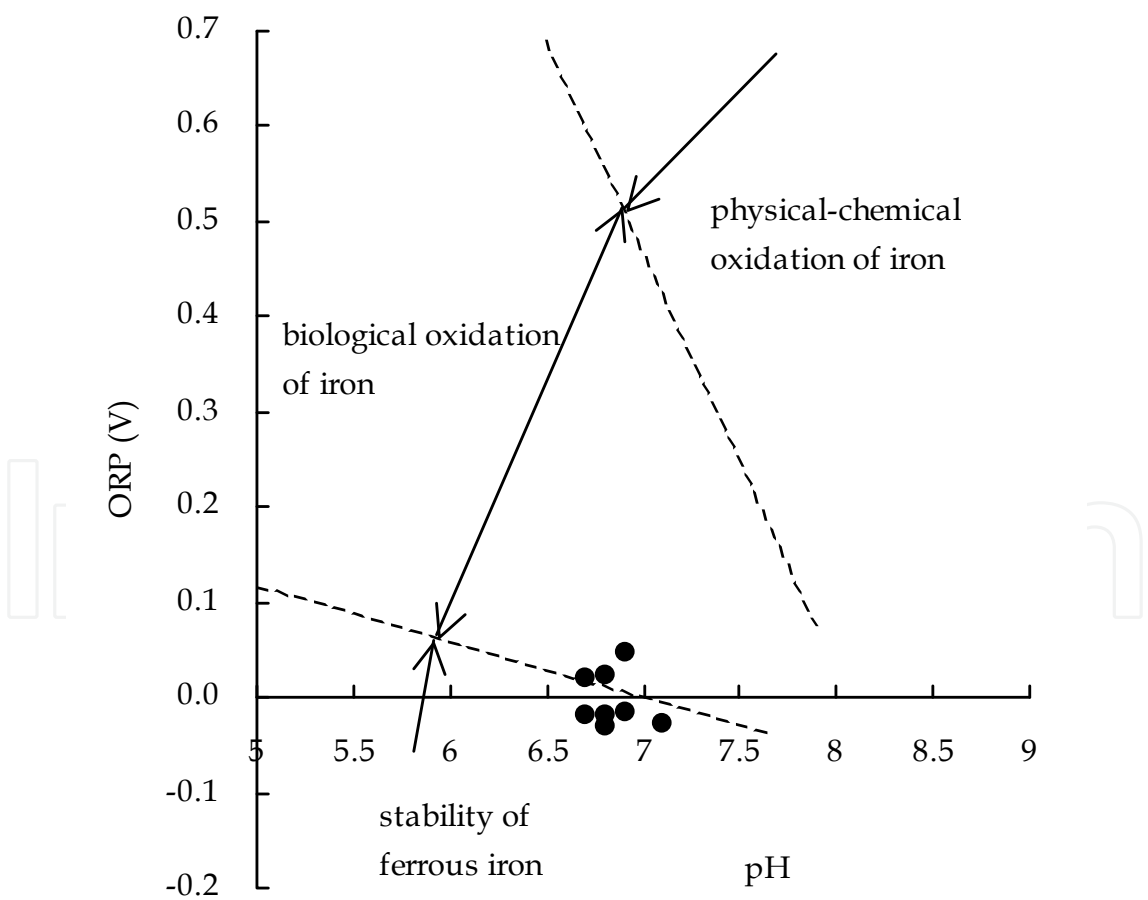


Fig. 7. Data plot on pH-OPR diagram (from Mouchet, 1992). Black dots represent the data monitored at the study sites of Fig. 4.



Fig. 8. Biogenic Fe oxides (brown mass) on woody carrier

Since the Fe oxidation was characterized with a pH-ORP diagram (Mouchet, 1992), the data from this study were plotted on it (Fig. 7). In this diagram, the pH-ORP area is divided into physical-chemical oxidation, biological oxidation, and stability of ferrous Fe. The data from this study were within the range of pH = 6.7 to 7.1 and ORP = -0.03 to 0.05 V, and were located near the boundary between the biological oxidation and the stable ferrous Fe area. Since the suitable aquatic conditions for the growth of Fe-oxidizing bacteria have been reported to be low concentration of oxygen and circumneutral pH (James & Ferris, 2004), the results of present study agree with this knowledge.

3.2 Fe and P on the carrier

The color on the woody carrier changed from light yellow to dark brown. Observation using a microscope revealed that biogenic Fe oxides produced by Fe-oxidizing bacteria had accumulated on the woody carrier (Fig. 8(a)). In many cases, the woody carriers were not easily visible because they had been completely covered by a mass of Fe oxides (Fig. 8(b)).

Figure 9 shows the Fe collected on the woody carrier and the D-Fe concentrations of the water at the site of the immersion test. The average accumulation of Fe on the Japanese cedar was 7.91 g/kg during the irrigation period and 6.74 g/kg during the non-irrigation period. The respective values for the Japanese cypress were 7.67 and 5.54 g/kg. There were no significant differences between the values during the irrigation and the non-irrigation period. The average D-Fe concentration during the irrigation period (0.952 mg/L) was much higher than that during the non-irrigation period (0.338 mg/L). There were no significant differences during the irrigation and non-irrigation period between the collected Fe for the Japanese cedar and the Japanese cypress (Fig. 10). When these values are expressed in parts per million (ppm), the Fe collected during the irrigation period was 7,910 ppm for the Japanese cedar and 7,670 ppm for the Japanese cypress, while the D-Fe concentration was 0.952 ppm. Therefore, the concentration of the Fe on the woody carrier was 8,000- to 8,300-fold greater than the Fe dissolved in the water. For the non-irrigation period, the degree of Fe concentration was 16,000- to 20,000-fold greater.

Figure 11 shows the P adsorbed on the woody carrier and the $\text{PO}_4\text{-P}$ concentration. The average P adsorbed on the Japanese cedar carrier was 0.350 g/kg during the irrigation

period and 0.187 g/kg during the non-irrigation period. The respective values for the Japanese cypress were 0.332 and 0.172 g/kg. The differences between the values during the irrigation and non-irrigation periods were significant ($p < 0.05$). The average $\text{PO}_4\text{-P}$ concentration of the water during the irrigation period (0.058 mg/L) was much higher than that during the non-irrigation period (0.022 mg/L). This is probably because the anaerobic conditions caused by flooded water on the paddy fields during the irrigation period lead to the reduction of ferric phosphate (FePO_4) compounds and the release of Fe^{2+} and phosphate (PO_4^{3-}) ions. There were no significant differences in the adsorbed P during the irrigation and the non-irrigation period between the Japanese cedar and the Japanese cypress (Fig. 12). When these values are expressed in ppm, the P adsorbed during the irrigation period was 350 ppm for the Japanese cedar and 332 ppm for the Japanese cypress, while the $\text{PO}_4\text{-P}$ concentration was 0.058 ppm. Therefore, the concentration of the P on the woody carrier was 5,700- to 6,000-fold greater than the P dissolved in the water, and for the non-irrigation period, it was 7,800- to 8,500-fold greater.

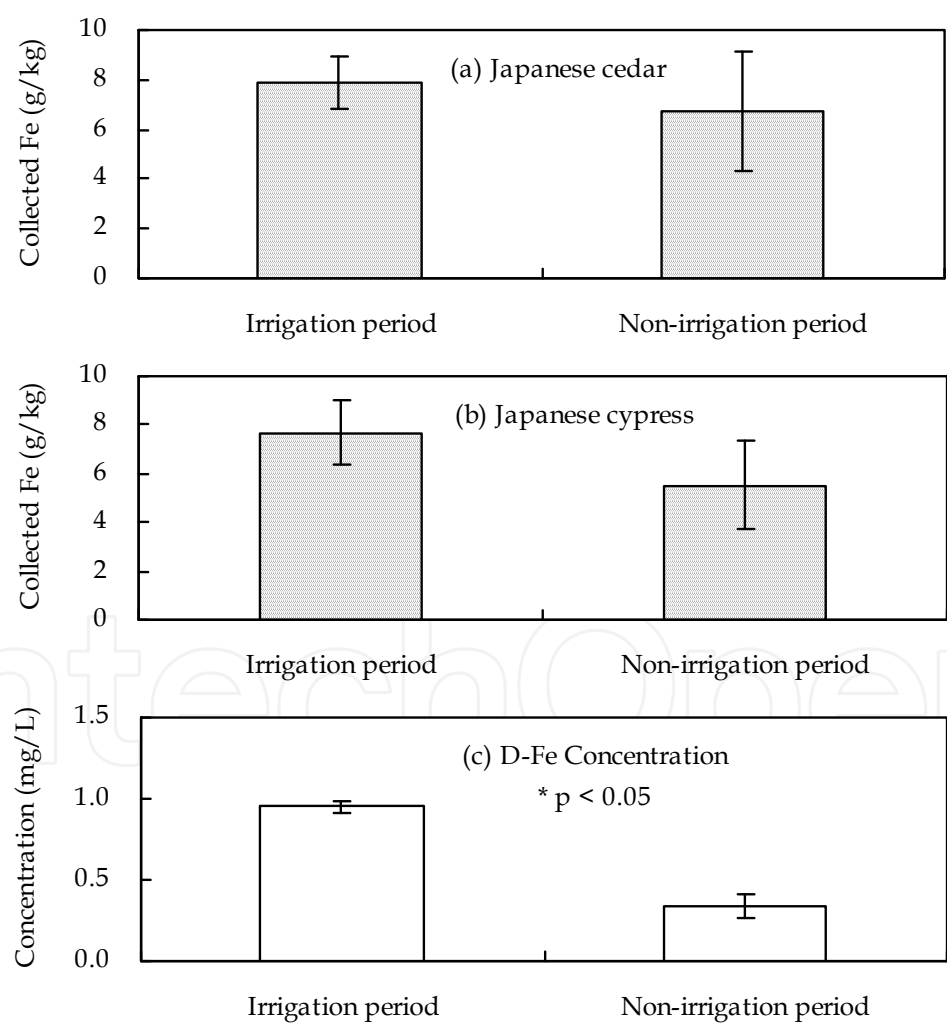


Fig. 9. Fe content after the immersion test. (a), (b): collected Fe after 4 weeks immersion; (c): D-Fe concentration of the water (means and standard errors, n=8)

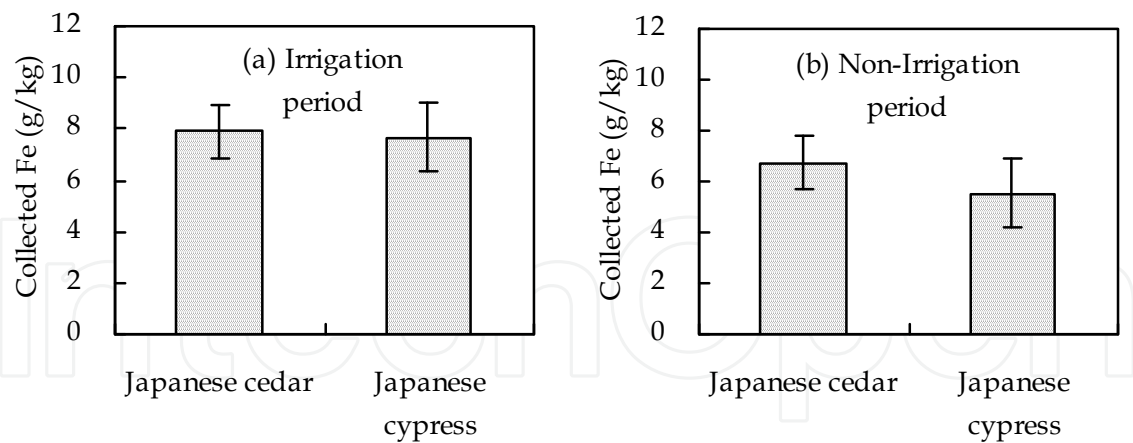


Fig. 10. Comparison of collected Fe between Japanese cedar and Japanese cypress (n=8)

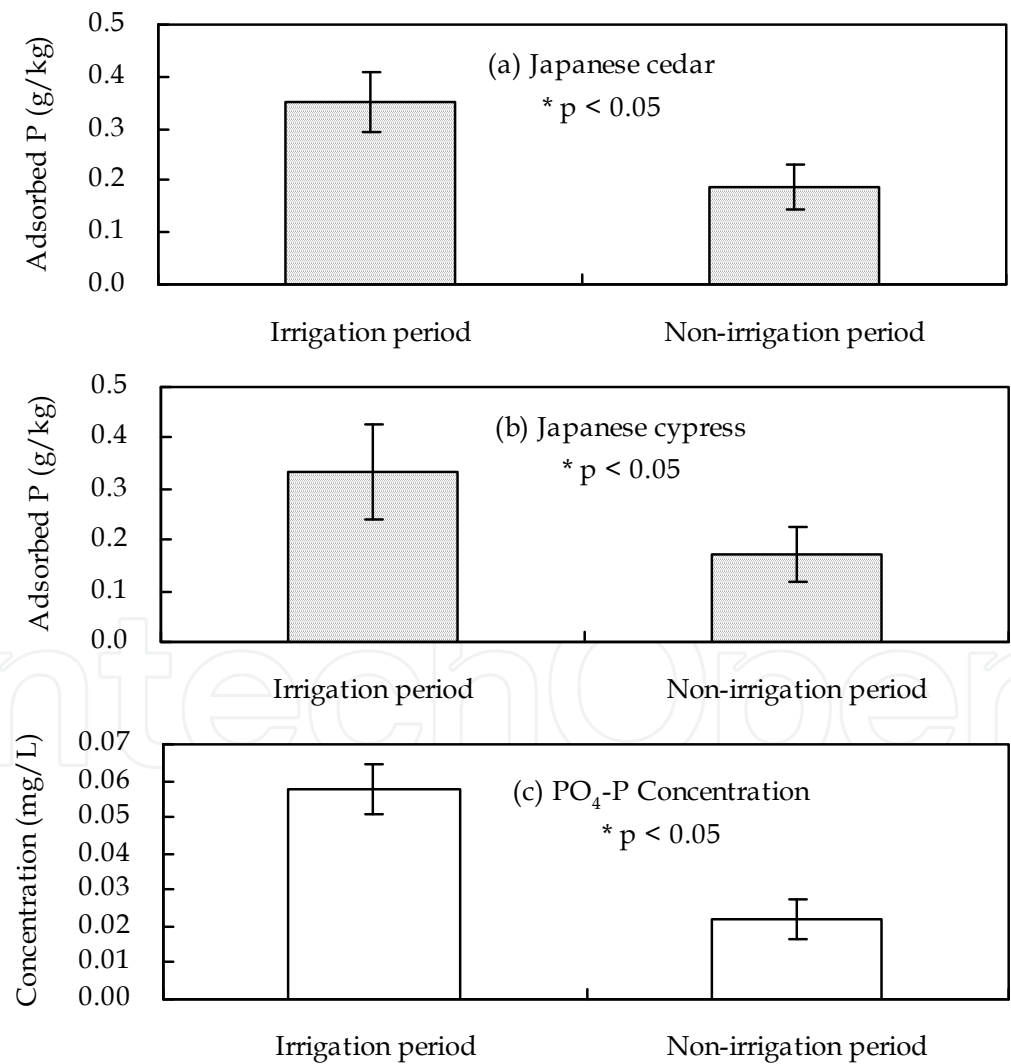


Fig. 11. P contents from the immersion test. (a), (b): adsorbed P after 4 weeks immersion; (c): PO₄-P concentration of the water (means and standard errors, n=8)

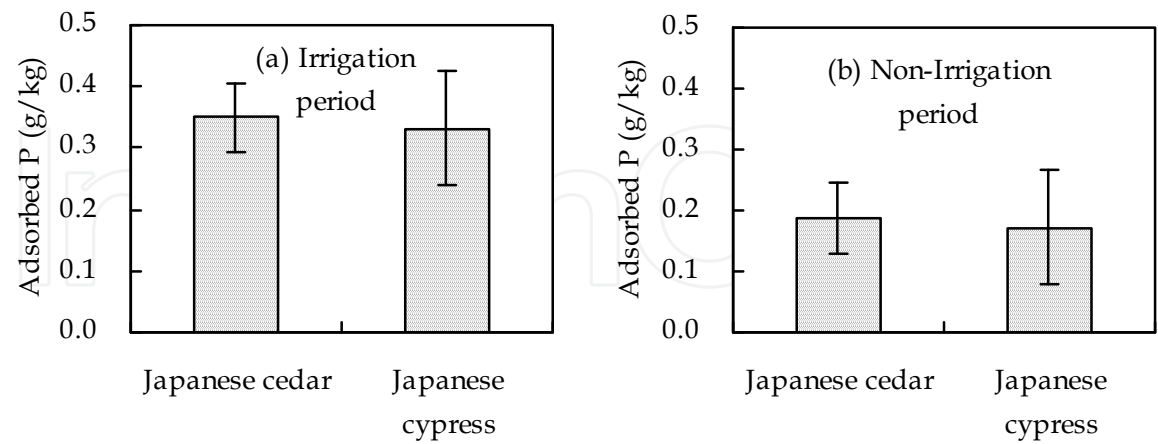


Fig. 12. Comparison of adsorbed P between Japanese cedar and Japanese cypress (n=8)

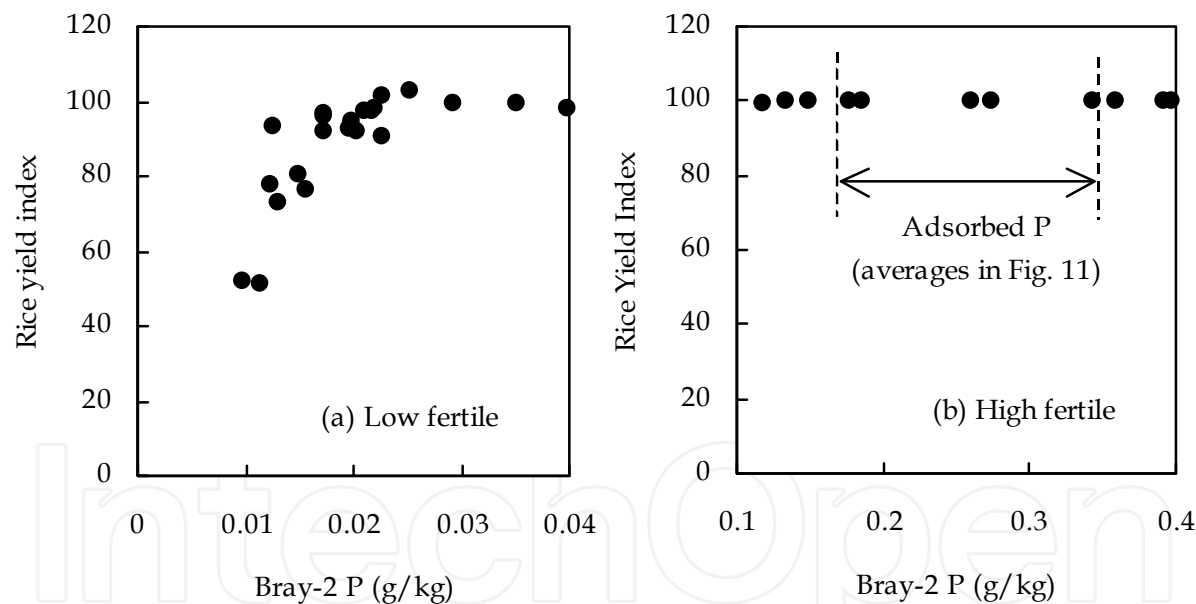


Fig. 13. P fertility of the immersed carrier in the relationship between the Bray-2 P in arable soils and the rice yield index (adapted from Komoto, 1984)

Figure 13 shows the P fertile position of the immersed carrier on the relationship between the Bray-2 P in arable soils and the rice yield index (adapted from Komoto, 1984). In low-fertility soil (Fig. 13(a)), the yield index increases with Bray-2 P, but does not increase over the fertile level of 0.025 g/kg of Bray-2 P. As shown in Fig. 13(b), soils containing greater than 0.1 g/kg are categorized as high-fertility soil. The P values from this study were between 8- and 17-fold higher than the required level (0.025 g/kg) and categorized in the range of high-fertility soil. Therefore, the immersed carrier had obtained sufficient P fertility.

3.3 Heavy metals on the carrier

Figure 14 shows an example of an X-ray fluorescence spectrum of the immersed carrier. Fe was the main species detected, although silicon (Si), calcium (Ca), aluminum (Al), P, sulfur (SO₄), potassium (K), chlorine (Cl) were also present. Heavy metals were not detected on most of the carriers, but traces of Pb and Zn were detected in some samples (Table 2). However, they were well below regulation levels set out in the Fertilizers Regulation Act (Ministry of Agriculture, Forestry and Fisheries, 2007) and the Guidelines against Heavy Metal Accumulation in Arable Soil (Environment Agency, 1984). This was probably because the study site was in a rural area that had not been contaminated by heavy metals and also because the immersion period was too short for these metals to accumulate.

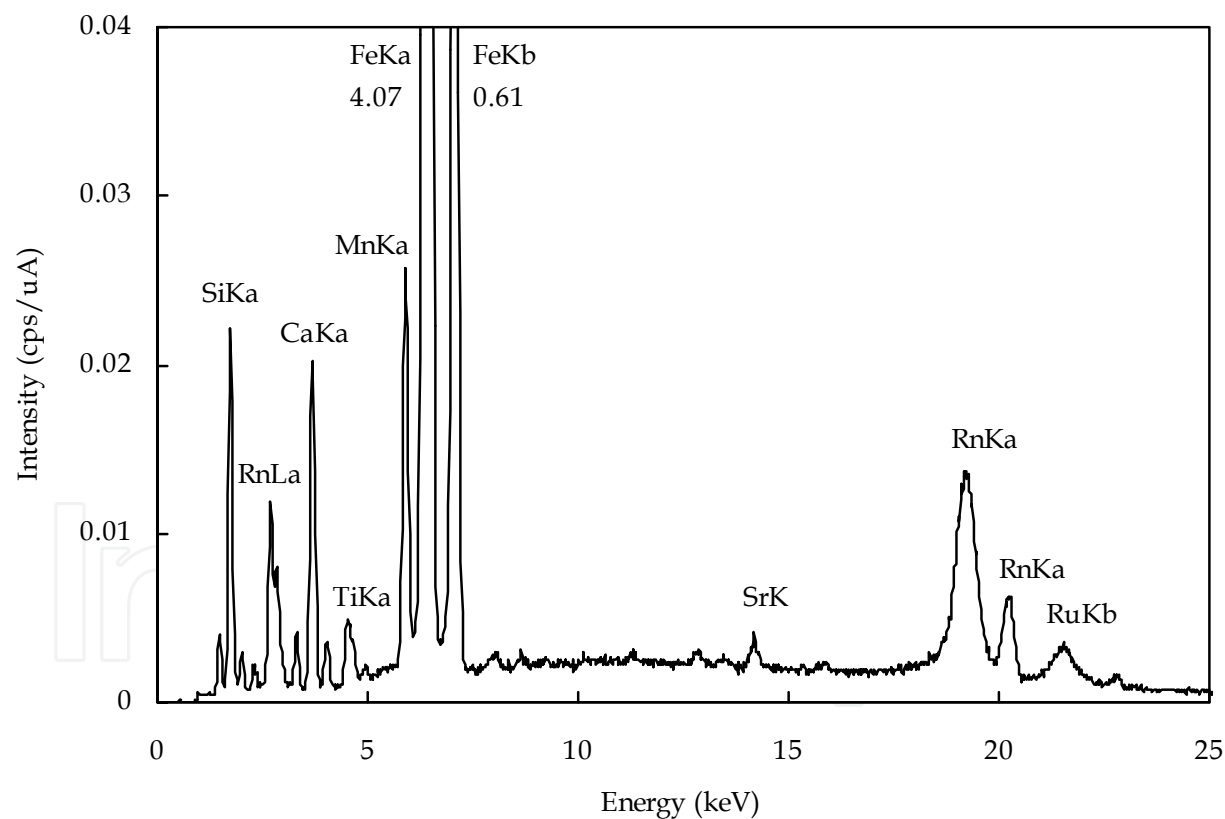


Fig. 14. X-ray fluorescence spectrum of immersed carrier

Element	Concentration (mg/kg)	Regulation value (mg/kg)
As	ND	50*
Cd	ND	5*
Cr	ND	500*
Hg	ND	2*
Ni	ND	300*
Pb	5.3	100*
Zn	4.0	120**
Cu	ND	125**

* Ministry of Agriculture, Forestry and Fisheries, 2007

** Environment Agency, 1984

Table 2. Heavy metal concentrations in immersed carrier (maximum for n=45)

3.4 Possible further applications

The findings reported in this chapter have been obtained from a specific region in Japan. However, Fe is the third most abundant metal found in the soil (Spark, 1995), and Fe-oxidizing bacteria are not rare (Emerson et al., 1999; Emerson & Weiss, 2004; James & Ferris, 2004). Thus, this method can be applicable in many places, provided suitable aquatic conditions supporting the growth of Fe-oxidizing bacteria (low concentration of oxygen and circumneutral pH) are available. In addition, the immersed woody carrier can be applied directly to agricultural land in the form of a fertilizer, without P extraction procedures, which are commonly required for P recovery methods. Therefore, this method is a low-cost technique that should contribute to P resource recycling and the improvement of the aquatic environment, if adopted on a large scale.

4. Conclusions

A new method of P recovery from natural water bodies using Fe-oxidizing bacteria and woody biomass (Japanese cedar and Japanese cypress) was applied in an agricultural canal during irrigation and non-irrigation periods. The amounts of P adsorbed on the carrier during these periods were 0.332–0.350 and 0.172–0.187 g/kg, respectively, while the PO₄-P concentrations of the water were 0.058 and 0.022 mg/L. Expressed these values in parts per million, the P adsorbed on the carrier was 5,700- to 8,500-fold more concentrated than the P dissolved in water. The P on the carrier was 8- to 17-fold higher than the required level for sufficient fertility to support rice production, and it was categorized in the range of high-fertility soil. Some traces of heavy metals adsorbed on the carrier were detected, but they were much lower than the regulation levels. In addition, the woody carrier can be applied directly to agricultural land without P extraction. Therefore, this method is a low-cost technique that should contribute to P resource recycling and the improvement of aquatic environment.

5. Acknowledgement

This study was partially supported by a grant from the Shimane University Priority Research Project and a Grant-in-Aid for Scientific Research from the Japan Society for the Promotion of Science (#20380179).

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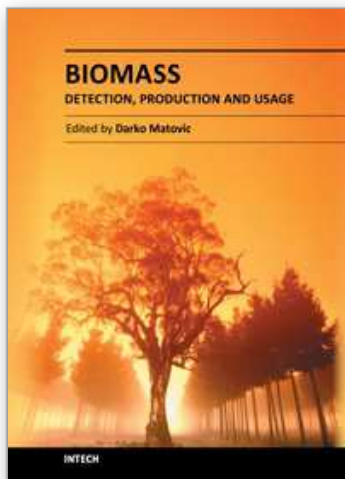
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Biomass - Detection, Production and Usage

Edited by Dr. Darko Matovic

ISBN 978-953-307-492-4

Hard cover, 496 pages

Publisher InTech

Published online 09, September, 2011

Published in print edition September, 2011

Biomass has been an intimate companion of humans from the dawn of civilization to the present. Its use as food, energy source, body cover and as construction material established the key areas of biomass usage that extend to this day. Given the complexities of biomass as a source of multiple end products, this volume sheds new light to the whole spectrum of biomass related topics by highlighting the new and reviewing the existing methods of its detection, production and usage. We hope that the readers will find valuable information and exciting new material in its chapters.

How to reference

In order to correctly reference this scholarly work, feel free to copy and paste the following:

Ikuo Takeda (2011). Recycling of Phosphorus Resources in Agricultural Areas Using Woody Biomass and Biogenic Iron Oxides, Biomass - Detection, Production and Usage, Dr. Darko Matovic (Ed.), ISBN: 978-953-307-492-4, InTech, Available from: <http://www.intechopen.com/books/biomass-detection-production-and-usage/recycling-of-phosphorus-resources-in-agricultural-areas-using-woody-biomass-and-biogenic-iron-oxides>

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