



A mobile pilot-scale plant for *in situ* demonstration of phosphorus recovery from wastewater using amorphous calcium silicate hydrates



Kenji Okano^{a,*}, Shimpei Miyamaru^a, Yasuhisa Yamamoto^a, Masashi Kunisada^b, Hiroyuki Takano^c, Masaya Toda^d, Kohsuke Honda^a, Hisao Ohtake^a

^a Department of Biotechnology, Graduate School of Engineering, Osaka University, 2-1 Yamada-oka, Suita, Osaka 565-0871, Japan

^b Mikuni Pharmaceutical Industrial Co., Ltd., 2-35 Kamisu-cho, Toyonaka, Osaka 561-0823, Japan

^c Research & Development Center, Taiheiyo Cement Co., 2-4-2 Osaku, Sakura, Chiba 285-8655, Japan

^d Research & Development Laboratory, Onoda Chemical Industry Co., Ltd., 39-13 Miyamoto-cho, Itabashi-ku, Tokyo 174-0054, Japan

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ABSTRACT

A mobile pilot-scale plant was developed for the *in situ* examination and demonstration of phosphate (P_i) recovery from wastewater. The mobile pilot plant consisted of a 1000-L reinforced-plastic reactor, a self-made filter, and ancillary equipment and was set up on a 1.5-tonne motor truck for transport. Five separate *in situ* experiments were carried out using the mobile pilot plant to evaluate the ability of amorphous calcium silicate hydrates (A-CSHs) to recover P_i from anaerobic sludge digestion liquor at a wastewater treatment plant. On average, approximately 80% P_i could be recovered from the anaerobic sludge digestion liquor by a process consisting of 20-min mixing, 30-min settling, and 90-min filtration. Approximately 20% of the dry weight of the recovered product was citrate-soluble P_2O_5 . The levels of heavy metals such as Cd, As, Pb, Ni, and Cr were much lower than their regulatory standards for fertilizer. The efficacy of the recovered product as P_i fertilizer was confirmed by plant cultivation tests using the leaf vegetable Komatsuna (*Brassica rapa* L. var. *perviridis*). The present study showed that the mobile pilot plant is useful as a simple, potentially low-cost tool for the *in situ* examination and demonstration of P_i recovery from wastewater.

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1. Introduction

Phosphorus (P) is one of the most critical elements in biological building blocks and plays an essential role in the cellular metabolism of all living organisms [9]. Since no other element can substitute for P in biochemical processes, humans ultimately rely on P availability. Today, P is mostly obtained from mined rock phosphate (P_i) [10]. However, natural reserves of high-grade rock P_i are limited and running out on a global scale [8]. Increasing attention has been paid to P recycling from secondary resources such as sewage sludge and animal manure to close the anthropogenic cycle of P [5].

Abbreviations: P, phosphorus; P_i , phosphate; A-CSHs, amorphous calcium silicate hydrates; NMR, nuclear magnetic resonance; WWTP, wastewater treatment plant; T-P, total-P; T-CO₂, total-CO₂; NH₄-N, ammonium nitrogen; A-CSHs-P, P_i adsorbed by A-CSHs; C-P₂O₅, citrate-soluble phosphate; T-K₂O, total potassium; T-N, total nitrogen; ANOVA, analysis of variance.

* Corresponding author.

E-mail address: okano@bio.eng.osaka-u.ac.jp (K. Okano).

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Although various technologies are potentially applicable to P_i recovery from wastewater [6,7,20], their implementation has often been economically infeasible because of the high cost of plant development, construction, and operation. To fully realize P recycling, it is necessary to improve the cost performance at various stages of development, including pilot-scale examination, plant construction, and full-scale operation.

Previously, we have reported a novel technique for P_i recovery from aqueous solution using amorphous calcium silicate hydrates (A-CSHs) [15,16]. A-CSHs can be synthesized using very widely available, inexpensive materials such as siliceous shale and Ca(OH)₂. ²⁹Si NMR analysis has revealed that A-CSHs consisted of silicate polymers (average chain length of 3.5) which were linked to each other through ion bindings with Ca²⁺ [15]. Importantly, A-CSHs could readily form Ca- P_i -silicate ion aggregates in water, thereby serving as a bifunctional adsorption-aggregation agent for P_i recovery.

To develop a new P_i recovery technology using A-CSHs, it is necessary to examine their performance at a pilot-scale level in a wastewater treatment plant (WWTP). However, it is obviously

costly and time-consuming to construct a pilot-scale plant at a WWTP merely for short-term *in situ* experiments. In addition, the construction of a pilot-scale plant in a WWTP often takes up more space than necessary and unavoidably hinders the regular service. A possible option for avoiding such inconvenience is to use a mobile pilot-scale plant that can be easily conveyed by a motor truck and readily installed at a WWTP for *in situ* experiments.

In the present study, we examined the ability of A-CSHs to recover P_i from anaerobic sludge digestion liquor at a WWTP using a mobile pilot-scale plant (Fig. 1). The mobile pilot plant consisted of a 1000-L reinforced plastic reactor, a self-made filter, and ancillary equipment. It was loaded on a 1.5-tonne motor truck and transported by road to the WWTP on the day of *in situ* experiments. The P_i recoverability by A-CSHs was evaluated in five *in situ* experiments using anaerobic sludge digestion liquor at the WWTP. The efficacy of the recovered P_i as a by-product P_i fertilizer was examined in plant cultivation tests.

2. Materials and methods

2.1. Mobile pilot-scale plant

A 1000-L reactor of 1.3-m height and 1.2-m diameter made of 1-cm-thick reinforced plastic was set up on a 1.5-tonne motor truck using a steel frame (Fig. 1). The lower part of the reactor, which had the shape of an inverted circular cone (130 L; 30° slope angle), was designed to collect P_i -rich solids by free sedimentation. The reactor was equipped with an agitator having two stirring blade (model KP4002B, Hanwa Kakoki Co., Osaka, Japan) for mechanical mixing. One inlet valve, which was fitted on the top of the reactor, was connected with an accordion hose (model TACSD-A, Totaku Industries Inc., Osaka, Japan) for inlet water. Another end of the accordion hose was connected to the outlet of a membrane-type solid-liquid separator for digested sludge in a full-scale WWTP. Anaerobic sludge digestion liquor, which was rejected water from the solid-liquid separator, was fed into the reactor through the accordion hose by gravity. Two outlet valves were fitted on the side of the reactor to drain the supernatant after the free sedimentation of P_i -rich solids. P_i -rich solids were withdrawn from the bottom of the reactor through an outlet valve. The inner diameter of the inlet and outlet valves was 5.0 cm. All valves were manually operated when needed. P_i -rich solids were filtered using a self-made filter system. To prepare the filter system, a 25-cm-diameter filter cloth tube (Public Sheet #200, Asahi Kasei Geotechnologies Co., Tokyo, Japan) was tied at one end with a flexible plastic band and hooked on a steel pipe frame. The filter cloth tube was made of high tenacity yarns of synthetic fibers, having a permeability of $5 \times 10^{-2} \text{ cm s}^{-1}$ and a thickness of 0.36 mm. Since the filter cloth tube is widely used for civil engineering work, it is tough, easy to handle and available at a cheap price. A 250-L plastic container was placed under the steel pipe frame to collect

the filtrate. This filtration system was set up on the ground beside the motor truck before being used.

2.2. *In situ* P_i recovery experiment

A-CSHs were chemically synthesized in a slurry form (73 g dry weight/L) at a Ca/Si molar ratio of 1.0 using a method described previously [16]. A-CSHs contained SiO_2 (42.0 mass%), CaO (39.4 mass%), Na_2O (8.5 mass%), and hydrated water (9.4 mass%) as main components. No toxic heavy metals were detected with the chemically synthesized A-CSHs. Moreover, A-CSHs contained no detectable amounts of $\text{Ca}(\text{OH})_2$ and CaCO_3 . Ca^{2+} was mostly bound to silicate polymers as has been shown previously [15]. The amorphous structure of A-CSHs has been shown by X-ray diffraction analysis [15].

In situ P_i recovery experiments were performed using the mobile pilot plant at a full-scale WWTP located in the Osaka area of Japan. This WWTP employs a Bio-P process (alternative anaerobic and aerobic activated sludge process) to remove P_i from wastewater. P_i -rich sludge is subjected to anaerobic digestion after being concentrated by centrifugation. Approximately 1000 L of anaerobic sludge digestion liquor was fed into the reactor through a self-made cotton filter to remove large sludge flocs. The typical composition of the anaerobic sludge digestion liquor used in the present study is shown in Table 1. The levels of total P (T-P), P_i , total CO_2 (T- CO_2) and ammonium nitrogen ($\text{NH}_4\text{-N}$) were typically 196, 175, 1280, and 607 mg/L, respectively (pH 7.9). Samples of about 5 mL were taken from the reactor and filtered through a 0.22- μm -pore-size Millex-GV filter (Merck Millipore, MA, USA). The P_i concentration was determined by the molybdenum blue method described by Murphy and Riley [12]. After measuring the initial P_i concentration, 8.1–9.8 L of A-CSHs slurry (590–720 g dry weight) was added to the reactor at a Ca/P molar ratio of 2.0. The dilution ratio of A-CSHs slurry to anaerobic sludge digestion liquor was 1:102–1:123. The reaction mixture was continuously stirred by a mechanical agitator at 200 rpm. The pH was monitored using a portable pH meter (D-51S, Horiba Ltd., Kyoto, Japan).

After 20–60 min of mixing, P_i adsorbed by A-CSHs was allowed to settle for 30–60 min. Then, approximately 830 L of the supernatant was drained from the reactor by operating the two outlet valves fitted on the side of the reactor (Fig. 1b). The remaining 170 L of water was withdrawn from the bottom of the reactor and poured into filter cloth bags. After 90 min of filtration, the filter

Table 1
Typical composition of anaerobic sludge digestion liquor.

Components (mg/L)							
T-P	P_i	T- CO_2	$\text{NH}_4\text{-N}$	Mg	Na	K	Fe
196	175	1280	607	7	50	47	N.D. ^a

^a Not detected.

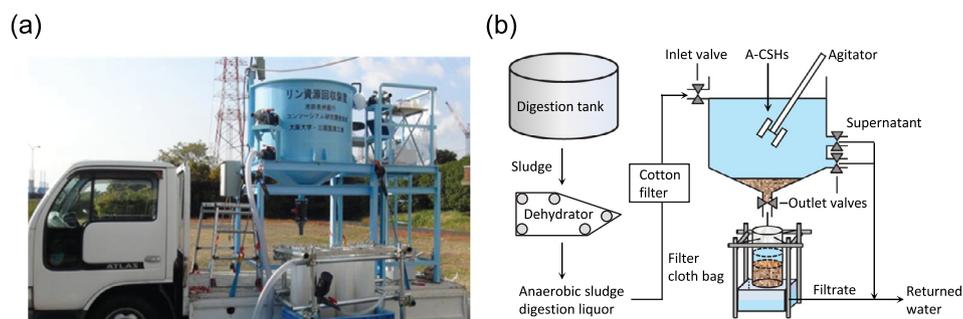


Fig. 1. Mobile pilot-scale plant (a) and schematic diagram of the P_i recovery process in *in situ* experiments (b).

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