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Effect of mineral elements on phosphorus release from heated sewage sludge

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Abstract

The aim of this work was to examine the influence of mineral elements on phosphorus (P) release from heated waste sludges. Energy dispersive X-ray microanalysis suggested that P was associated with Al, Ca, and Mg on the surface of waste sludge biomass obtained from six wastewater treatment plants. The extent of P release decreased with increasing the total concentrations of Al, Mg, and Ca in waste sludges. The addition of $Al_2(SO_4)_3$, $Ca(OH)_2$, $CaCl_2$, $MgSO_4$, or $NaAlO_2$ to activated sludges, which were taken from a bench-scale EBPR process, reduced the P release significantly.

Keywords: Activated sludge; Mineral elements; Phosphorus recovery; X-ray microanalysis

1. Introduction

Phosphorus (P) is an irreplaceable constituent for all types of living organisms. Natural ore deposits containing high-quality P are limited and they are rapidly dissipating (Ableson, 1999). Since the current major use of P is in fertilizers, the reuse and recycling of P are of great importance for sustaining profitable agricultural production in the long term (Kuroda et al., 2002). On the other hand, increased input of inorganic phosphate (P_i) to lakes, bays, and other surface waters causes eutrophication, resulting in excess growth of phototrophs, depletion of dissolved oxygen, degradation of the recreational value of water, and appearance of foul tastes in drinking water (Hammond, 1971). Since eutrophication of water bodies is a major worldwide problem (de-Bashan and Bashan, 2004), substantial attention has been paid to the efficient removal of P_i from wastewater (Ohtake et al., 1985).

Enhanced biological P removal (EBPR) is a well-established process that has been practiced widely in full-scale wastewater treatment facilities (Sedlak, 1991). In EBPR processes, sludge microorganisms accumulate excess P_i in the form of polyphosphate (polyP), which is a linear polymer of P_i residues linked by high-energy phosphoanhydride bonds (Fuhs and Chen, 1975). Waste sludges generated by the EBPR processes contain large amounts of P and thus may be a viable source of P for recycling as a fertilizer or an ingredient in other valuable P products (de-Bashan and Bashan, 2004).

Kuroda et al. (2002) proposed a simple technique for recovering P in a reusable form from polyP-rich activated sludge. The principle of the proposed technique was based on the finding that polyP could be released from activated sludge simply by heating it at 70 °C for approximately 1 h followed by precipitation upon the addition of $CaCl_2$ at room temperature. The product of this process was hydroxyapatite ($Ca_5(PO_4)_3OH$) containing low amounts of organic compounds and metal cations (Mg, Fe, Al). In many wastewater treatment plants (WTPs), waste sludge is subjected to anaerobic digestion in order to generate

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methane gas, which can potentially be used for heating the sludge to release polyP. It is also possible to employ a heat exchanger to further reduce heating energy consumption (Kuroda et al., 2002). More recently, Takiguchi et al., (2003) demonstrated that this method was applicable to P recovery from waste sludges in a pilot plant-scale EBPR process. However, the extent of P release from waste sludges was significantly lower in the pilot-scale EBPR process than that observed in laboratory experiments. Since waste sludges generated by WTPs contain a variety of mineral elements such as Al, Ca, Fe, and Mg (Schoenborn et al., 2001), which can form poorly soluble complex compounds with P, it is likely that the presence of these elements negatively affects the P release from heated waste sludges. In the present study, the influence of elements such as Al, Ca, Fe, and Mg on P release from heated waste sludges was examined.

2. Methods

2.1. Heat treatment and P precipitation

The bench-scale EBPR process used in the present study was the same as described previously (Kuroda et al., 2002). Laboratory activated sludges (L-sludges) were obtained from the EBPR process which had been stably maintained for at least one month. The sludge concentration in the EBPR process was maintained at approximately 2.5–3.2 g/L. Settled, fresh return-activated sludges were collected from six different WTPs (Hiroshima, Japan), none of which employed an EBPR process. They were designated WTP-sludges. The sludge concentrations were in the range of 3.8–6.7 g/L. Sludge samples were heated at 70 °C for 1 h using a temperature-controlled water bath as described previously (Kuroda et al., 2002). Heated samples were then centrifuged at 8000*g* for 5 min. For P precipitation, $CaCl_2$ was added to the supernatant at the stoichiometric Ca:P ratio of 1:1 (pH 11). After 2 h of incubation at room temperature, the precipitates were collected by centrifugation at 8000*g* for 5 min.

2.2. SEM and X-ray microanalysis

The scanning electron microscope (SEM) (JSM-5900, JEOL) was used to take microscopic pictures of sludge microorganisms. Samples were fixed with 2.5% glutaraldehyde at 4 °C for 2 h and then dehydrated in a graded ethanol series. Samples were dried overnight at −20 °C and then coated with carbon (JEC520 Carbon Coater, JEOL). Samples were viewed at an accelerating voltage of 20 kV, and the images were captured by digital mapping software (EX-55020, JEOL). The SEM was equipped with a spectrometer (JED-2200, JEOL) capable of detecting X-rays emitted by the specimen during electron-beam excitation. Energy-dispersive X-ray (EDAX) analysis was performed using the JED-2200 spectrometer at an accelerating voltage of 20 kV.

2.3. Analytical methods

Intracellular P_i was extracted from sludge biomass by cold 10% trichloric acid (TCA) for 30 min. The cold TCA extract also contained P_i which was removed from the surface of sludge biomass by TCA. P_i was determined by the ascorbic acid method after pH adjustment (Clesceri et al., 1989). PolyP was determined by hydrolysis in 1 N HCl at 100 °C for 7 min (Harold, 1960). Total phosphorus (T-P) was assessed as P_i after ammonium persulfate digestion at 120 °C for 30 min (Clesceri et al., 1989). The total concentrations of Mg, Al, and Ca in waste sludge samples were determined using Inductively Coupled Plasma Emission Atomic Spectrometry (ICP-AES) (SPSs 3000, Seiko Instruments) after digesting the samples in concentrated nitric and hydrochloric acids.

2.4. Statistical analysis

The statistical analysis was performed with SPSS7.5 (SPSS Inc., Chicago, Illinois, USA). The student's *t*-test was used to compare the changes in P contents of L- and WTP-sludges before and after heat treatment. Differences were considered statistically significant at a p value of <0.05. Regression analysis was used to determine the relationships between P and mineral elements. Analysis of variance (ANOVA) was also conducted at $\alpha = 0.05$ and 0.01 to detect the significant effect of mineral salt addition on the P release from heated L-sludges.

3. Results and discussion

3.1. P release by heat treatment

Fig. 1 depicts the typical data on P release from L- and WTP-sludges. L-sludges accumulated large amounts of polyP (approximately 3.5–5.0% as P of their dry weight). The T-P contents varied from 4.3% to 6.9% on a dry weight basis. The release of T-P and polyP from the L-sludges by heating averaged 70% and 85%, respectively. For WTP-sludges, polyP was present at 1.2–2.3%, and T-P was from 2.6% to 4.4% on a dry weight basis (Table 1). The relatively low P contents of the WTP-sludges could be attributed to the fact that none of the six WTPs employed an EBPR process. There was also a significant deference in P_i levels between L- and WTP-sludges. As shown in Fig. 1, the higher levels of P_i were detected with WTP-sludges. Since the levels of intracellular P_i could be considered essentially constant (Ohtake et al., 1985), this result suggested that significant amounts of P_i were present on the surfaces of WTP-sludges. The extent of P release from the WTP-sludges was highly variable, ranging from 34% to 56% of T-P (Table 1). This was unlikely due to the different levels of T-P and polyP in the WTP-sludges, since no significant relationship was found between the P release and the P content of waste sludges (Table 1). The addition of $CaCl_2$ precipitated 74–87% of the released T-P, resulting in T-P