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In situ and short-time anaerobic digestion coupled with alkalization and mechanical stirring to enhance sludge disintegration for phosphate recovery

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HIGHLIGHTS

- Anaerobic digestion coupled with alkalization and mechanical stirring was studied.
- Initial pH pretreatment was effective for disintegration with greater SOP release.
- Chemical effects has a more significant role than bacterial ones with pH increase.
- Alkalization transformed all NAIP and most of the IP into phosphate in supernatant.

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ABSTRACT

In situ and short-time anaerobic digestion coupled with alkalization and mechanical stirring was studied to enhance sludge (especially high-inorganic sludge) disintegration for phosphate recovery. Anaerobic digestion batch experiments with varying conditions were carried out. It was observed that high initial pH values (e.g. 9, 10, 12 and 13) in conjunction with mechanical stirring resulted in effective sludge disintegration. Results showed that mechanical stirring mainly worked as the role of uniform mixing for better material exchange and bacteria contact, but without the function of vortex shear force for cell wall breaking. Bacterial effects contributed less and less to phosphate recovery from sludge as alkalization increased, indicating that chemical effects have a more significant role during operation. Meanwhile, almost all insoluble non-apatite inorganic phosphorus was dissolved back into the supernatant with alkalization and mechanical stirring. Therefore, compared with other sludge disintegration methods, in situ and short-time anaerobic digestion coupled with alkalization and mechanical stirring is economical and practical, requiring only 7 days for sludge disintegration, especially with optimum alkalinity (pH 9 & 10).

1. Introduction

Waste sludge is the biggest waste with volume generated daily by activated sludge-based wastewater treatment plants (WWTPs). Most countries dispose waste sludge to landfill sites after mechanical dewatering to above 20% solid content [1–3]. However, landfill has long been recognized as an unsustainable practice, with waste sludge disposal becoming an increasingly expensive option especially in land-scarce metropolis, such as Auckland, Hong Kong, Shanghai, and Shenzhen [4,5]. As quantified in Australia and Europe, where the cost of waste sludge treatment can reach to about \$150–350/ton and \$165–550/ton, respectively [1]. Due to an unmatched urbanization process in China, over 3000 new WWTPs were built over the past few years, and as a result over 34 million tons of waste sludge is produced yearly with a projected 13% annual increase [1,6,7]. Although the disposal cost can vary significantly in different areas of China, the total added cost is still high. Therefore, effective methods carried out for waste sludge treatment, and phosphate recovery is considered as a positive and environmentally-friendly technology, required for waste sludge.

Phosphate recovery from waste sludge can be classed into two steps: sludge disintegration and phosphate precipitation [8]. Magnesium ammonium phosphate precipitation (MAP, also called struvite crystallization, Eq. (1)) is currently considered as the most economical and mature technology for phosphate precipitation.

$$Mg^{2+} + NH_4^+ + PO_4^{2-} + 6H_2O \rightarrow Mg(NH_4)PO_4{\cdot}6H_2O \quad (1)$$

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However, sludge disintegration is still an obstacle for the phosphate recovery process. The sludge disintegration enhancement process is considered as a key step for effective phosphate recovery from sludge [9,10]. As previous reviews have reported [11,12], numerous methods for sludge disintegration have been carried out, such as: acid and alkaline treatment, ozonation, pre-oxidation, thermal treatment, high pressure homogenizer, ultrasonication, electric pulse power method, gamma irradiation, stirred ball mills, lysate-thickening centrifugal technique, jetting and colliding method, microwave treatment, and anaerobic digestion. Some of these technologies however, are still in the lab scale phase due to economical and practical reasons, making anaerobic digestion the preferred method for sludge disintegration. But, anaerobic digestion has the disadvantages of long sludge rotation time (approximately 15–30 days) and it requires complex equipment, hindering its nationwide application for China. Therefore, traditional anaerobic digestion becomes unsuitable for high-inorganic sludge, and most WWTPs in Shenzhen choose to forgo the anaerobic digestion process for waste sludge. Moreover, in the south of China, especially in Shenzhen, over 50% of the sludge remains inorganic after chemical treatment with Al^{3+} or Fe^{3+} for phosphate removal. Additionally, though Al^{3+} or Fe^{3+} can effectively remove phosphate from wastewater, formation of precipitates ($AlPO_4$ and $FePO_4$) normally contains plenty of toxic impurities. Comparatively, struvite phosphate recovery from manure, is capable of precipitating toxic impurities, hence it is generally considered as the best method for recycling waste sludge [8].

In recent literatures, anaerobic digestion with alkalization has been widely studied and considered as a positive method for sludge treatment [13,14]. Alkalization can hydrolyze and decompose lipids, hydrocarbon, and proteins into smaller soluble substances. The process also causes cell wall damage [15], and further promotes the death of aerobic bacteria [16,17]. However, maintaining a stable alkaline condition for anaerobic digestion still has the problem of high running costs. Therefore, this study aims to identify a positive method for economical anaerobic digestion.

In situ and short-time (7 days) anaerobic digestion coupled with alkalization and mechanical stirring was carried out in this study to enhance sludge disintegration for phosphate recovery. Mechanical stirring is directly related to operational cost. Identifying the function of mechanical stirring could improve the mechanism understanding of anaerobic digestion with alkalization and mechanical stirring, and further optimize the operational cost. Moreover, identifying the function of mechanical stirring also could be benefit to study the role of alkalization in this study. Consequently, this study focused on the mechanism and function of the alkalization and mechanical stirring, and pointed out the significant roles involved in mechanical stirring for anaerobic digestion. In addition, the economical and practical analysis of this method was carried out in this study.

2. Materials and methods

2.1. Waste sludge sample

The waste sludge sample was obtained from the sludge equalization tank after the secondary sedimentation tank of Binhe municipal wastewater treatment plant in Shenzhen, China. Binhe WWTP serves the majority of downtown Shenzhen (27.5 km^2) with a population of 540 thousand people, it has a capacity of 300,000 m^3/d and is equipped with anaerobic-anoxic-oxic processes. The effluent of Binhe WWTP reaches 15 mg/L of TN and 0.5 mg/L of TP. The characteristics of the waste sludge used in this study are shown in Table 1.

2.2. Mechanical stirring batch experiment

Mechanical stirring batch experiments were carried out with two sets of anaerobic digestion reactors (ADR) (three replicate reactors for each set). ADR (Fig. S1) was covered with aluminum-foil paper for the inhibition of phototrophic bacteria. Each ADR was filled with 500 mL waste sludge. One set of ADR (ADR-MS) was mechanically stirred with a magnetic stirring apparatus, another set (ADR-SD) was anaerobically digested statically. Stirring rate of ADR-MS was controlled in the range of 400 rpm to ensure no vortex on the mixed liquid surface for the anaerobic condition. The experiment was operated for 7 days. The supernatant was collected from the mixed liquid after centrifugation at 700 rpm for 1 min. Each item of mixed liquid and its supernatant was measured as mentioned in the following.

2.3. Anaerobic digestion reactor (ADR) with alkalization and mechanical stirring batch experiment

Alkalization and the mechanical stirring batch experiment was applied with five sets of ADR (three replicate reactors for each set). As Zheng et al. [18] reported, pH tolerance of alkaliphilic microorganisms was in the range of 9–10, and the optimum pH for short-chain fatty acid (SCFA) release of sludge was 10. Hence, optimum alkalinity (pH = 9 and 10), excess alkalinity (pH = 12 and 13) and control alkalinity (no alkalinity adjustment, that was ADR-MS) were applied with 5 set ADRs (Fig. S1) in this study. For each ADR, 500 mL excess sludge was filled, and adjusted with NaOH (50 g/L) to a corresponding value. Then, ADR was stirred with 400 rpm for 7 days, which was similar as the mechanically stirred batch experiment. The supernatant was collected from the mixed liquid after centrifugation at 700 rpm for 1 min. Each item of mixed liquid and its supernatant was measured. In addition, the batch experiment of ADR with only alkalization was also carried out in this study.

2.4. Alkaline phosphatase activities in different pH conditions

Alkaline phosphatase (PO_4ase) measurement of activated sludge was carried out with an alkaline phosphatase kit for soil (S-AKP/ALP, Solaribio, Beijing, China). A portion of 1 mL sludge sample from an ADR was centrifuged at 8000 × *g* for five minutes. The supernatant was then discarded. The remaining sludge pellet was used to carry out the phosphatase measurements according to the manufacture's recommendation.

2.5. Analytical methods

Ammonia nitrogen (NH_4^+-N), total phosphate (TP), total nitrogen (TN), mixed liquor suspended solid (MLSS) and mixed liquor volatile suspended solids (MLVSS) were measured in accordance with the Standard Methods [19]. Total phosphate (TP) of samples was measured with molybdenum-antimony anti-spectrophotometric method with $K_2S_2O_8$ digestion pretreatment at 120 °C for 30 min in an autoclave (LT-CPS38C, Lead-Tech Scientific Instrument Co. Ltd., China). Soluble phosphate (SOP, which is also the recoverable form of phosphate) of liquid samples was analyzed with molybdenum-antimony anti-spectrophotometric method with pre-filtration (0.45 μm filter). Total organic carbon (TOC), polysaccharide, protein and nucleic acid were analyzed with TOC analyzer (Multi N/C 3100, Analytic Jena, German), phenol-sulfuric acid method, Coomassie G-250 method, and diphenylamine colorimetric method, respectively. The sludge samples after digestion were measured with SEM (scanning electron microscopy; ZEISS SUPRA® 55, Zeiss, German)-EDX (energy-dispersive X-ray analyzer; Oxford Isis, UK) with liquid N_2 freeze and air dry pretreatment. All items of the supernatant were immediately filtered through 0.45 μm filter (Anpel, Shanghai, China) before analysis. P fractionations in the solid phase of mixed liquid was analyzed according to Zhu et al. [20] (detailed in Supporting information (SI)). Measurements of SCFA and person's product momentum correlation coefficient (r_p) were also mentioned in SI.