

Calcium addition to increase the production of phosphate granules in anaerobic treatment of black water

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ARTICLE INFO

Article history:

Received 23 August 2017

Received in revised form

7 November 2017

Accepted 8 December 2017

Available online 8 December 2017

Keywords:

Phosphate recovery

Calcium phosphate

Anaerobic treatment

Black water

UASB reactor

ABSTRACT

Simultaneous recovery of calcium phosphate granules (CaP granules) and methane from vacuum collected black water (BW), using an upflow anaerobic sludge blanket (UASB) reactor was previously investigated. It was calculated that only 2% of the total phosphorus (P) fed was present as CaP granules whereas 51% of the P accumulated dispersed in the reactor, limiting the applicability of this process for recovery of phosphate. This study proposes adding calcium to increase the P accumulation in the reactor and the production of CaP granules. Calcium was added in a lab-scale UASB reactor fed with BW. An identical UASB reactor was used as reference, to which no calcium was added. The treatment performance was evaluated by weekly monitoring of influent, effluent and produced biogas. Sludge bed development and CaP granulation were assessed through particle size analysis. The composition and structure of CaP granules were chemically and optically assessed. Calcium addition increased accumulation of P in the reactor and formation and growth of granules with size > 0.4 mm diameter (CaP granules). Moreover, with calcium addition, CaP granules contained 5.6 ± 1.5 wt% of P, while without calcium a lower P content was observed (3.7 ± 0.3 wt%). By adding Ca, 89% of the incoming P from BW accumulated in the reactor and 31% was sampled as CaP granules (> 0.4 mm diameter). Addition of 250 mgCa L^{-1} of BW was the optimum loading found in this study. Furthermore, no significant reduction in $\text{COD}_{\text{Total}}$ removal (> 80%) and CH_4 production ($0.47 \pm 0.10 \text{ gCOD-CH}_4 \text{ g}^{-1}\text{COD}_{\text{Total-BW}}$) was observed. Therefore, adding calcium can significantly increase the CaP granulation without inhibiting the simultaneous CH_4 recovery. This further indicates the potential of this process for phosphate recovery.

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1. Introduction

Recovery of phosphate from wastewater is crucial for the sustainable use of resources (Cordell et al., 2011). European regulation requires removal of phosphorus (P) from wastewater (Schröder et al., 2010), due to the severe environmental impact of P discharge in natural water courses (Jarvie et al., 2006). On the other hand, the processing and extraction costs of natural phosphate rock (P rock), which is the present main source of P, are significantly increasing due to the decrease in ore quality and its geographic

distribution (Aydin et al., 2010; Koppelaar and Weikard, 2013). Thus, the need to remove P from wastewater and the increasing concerns about the future availability of P, have lead many researchers to focus on developing processes to recover phosphate from wastewater streams.

By vacuum collection of source separated black water (BW), which uses 1 L of water per toilet flush, concentrations of total P ranging from 0.15 up to 0.22 gP L^{-1} of BW are obtained (Graaff et al., 2011). Measurements show that in The Netherlands up to $480 \text{ gP person}^{-1} \text{ year}^{-1}$ could potentially be recovered, considering a BW production of $6 \text{ L person}^{-1} \text{ day}^{-1}$ (Kujawa-Roeleveld and Zeeman, 2006; Tervahauta et al., 2013). If this number is extrapolated for the global population, the phosphate recovery from BW would represent up to 14% of the current global demand of phosphate as P rock (Cooper et al., 2011; U.S. Geological Survey, 2015). The anaerobic treatment of BW using an upflow anaerobic sludge blanket

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(UASB) reactor has been tested at full scale since 2006 in Sneek (The Netherlands) (Graaff and Hell, 2014). At 25 °C and hydraulic retention time (HRT) of 8 days, 54% of the chemical oxygen demand (COD) in BW is effectively converted into methane (CH₄) and 61% of the total incoming P leaves the reactor in the effluent mostly as soluble phosphate (PO₄³⁻) (Graaff et al., 2010).

A new process for simultaneous recovery of calcium phosphate granules (CaP granules) and CH₄ in a UASB reactor treating BW was proposed by Tervahauta et al. (2014). This proposal was based on observed calcium phosphate granulation (CaP granulation) which started after an increase in soluble calcium (Ca²⁺) in BW, triggering the formation of seed particles that served as initial nuclei for microbial colonization and calcium phosphate (Ca_x(PO₄)_y) precipitation (Tervahauta et al., 2014). As a result, the accumulation of P in the UASB reactor increased from 39 to 51% (Graaff et al., 2011; Tervahauta et al., 2014). However, only 2% of the total incoming P from BW was present as CaP granules after 988 days (Tervahauta et al., 2014), limiting the applicability of combined recovery of CaP granules and CH₄ from BW.

Previous research has shown that Ca²⁺ addition in the effluent of a UASB reactor treating BW increased P precipitation (Cunha et al., 2017). Therefore, this study proposes adding Ca²⁺ directly in the reactor and the production of CaP granules, without significantly affecting the conversion of COD into CH₄. This was experimentally investigated by running two identical lab scale UASB reactors fed with BW under similar operational conditions, except for the Ca²⁺ loading.

2. Materials and methods

2.1. Experimental set-up

The experimental setup consisted of two identical glass UASB reactors (5.1 L) fed with BW as shown in Fig. 1. BW was collected from a neighborhood equipped with vacuum toilets in Sneek (The Netherlands) and stored at 7 °C prior use (Graaff et al., 2010). The inoculum (2 L) was taken from the UASB reactor described in Graaff and Hell (2014) and sieved before inoculation to remove particles larger > 0.4 mm diameter. Both reactors were operated for 460 days. Reactor R1 served as a control, while reactor R2 was dosed with CaCl₂·2H₂O to test the influence of Ca²⁺ addition P accumulation and extent of CaP granulation. The overall operational period was divided in two phases. Phase 1 lasted from day 0 to 350 and it was considered as the start-up and adaptation period of the sludge inoculum. About once a month sludge was removed to keep the sludge bed volume approximately at 70% (3.5 L) of the total working volume of the reactor. During the first 150 days of phase 1 the concentration of Ca²⁺ addition increased gradually every 50 days by steps of 100 mg L⁻¹_{BW} starting from 250 mg L⁻¹_{BW}. After 150 days the addition of Ca²⁺ decreased to 200 mg L⁻¹_{BW} until phase 2 (350 days), in order to decrease the saturation of Ca²⁺ in the reactor. Phase 2 lasted from day 350 to 460. During phase 2, the dosed Ca²⁺ loading in R2 was gradually increased once more from 150 mg L⁻¹_{BW} on day 350 to 200, 250, 300 and 400 mg L⁻¹_{BW} on day 370, 390, 420 and 440, respectively. Sludge bed sampling during phase 2 was performed on days 350, 415, 436 and 460, and the particle size distribution and elemental composition of each size fraction was measured to evaluate the formation and growth of CaP granules. Influent and effluent were continuously monitored, at least once a week, for: total and volatile suspended solids (TSS and VSS, respectively); total, colloidal and soluble COD (COD_{Total}, COD_{Colloidal} and COD_{Soluble}, respectively); total, organic and inorganic carbon (TC, TOC and IC, respectively); volatile fatty acids (VFA); ammonium (NH₄⁺); anions (Cl⁻, NO₃⁻, NO₂⁻, SO₄²⁻ and PO₄³⁻) and total and soluble

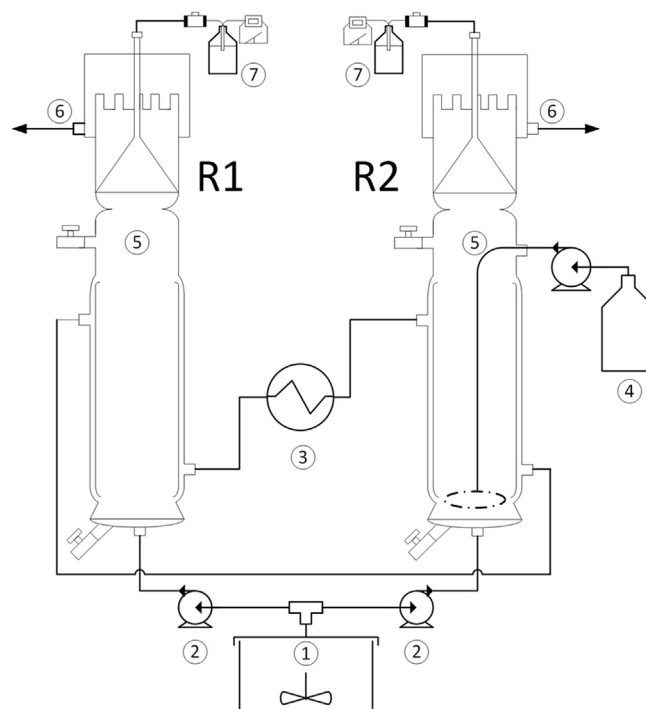


Fig. 1. Scheme of experimental setup: ① Storage tank of black water, cooled (7 °C) and stirred; ② Feeding pumps (Masterflex L/S 77200-60); ③ Shared heating system (Thermo Scientific, DC10 KC10) for temperature control (25 °C); ④ CaCl₂·2H₂O feeding system for reactor R2; ⑤ UASB reactors (5.1 L) with 9.7 cm diameter, 69 cm height and double glassed walls; ⑥ Effluent point after the gas-liquid-solid (GLS) separator; ⑦ Gas sampling and gas counter (Ritter MGC-1V3.3 PMMA).

elements (P, Ca, Mg, K and Na). Biogas production and composition were monitored with the same frequency.

2.2. Physico-chemical analysis

The influent flow rate was determined by effluent collection during approximately 24 h. Analyzed effluent was not older than one day and BW samples (0.5 L) were taken from the storage tank. TSS and VSS concentrations were determined by gravimetric standard method (Clesceri et al., 1998). COD_{Total} was determined by Hach Lange kits LCK014 from the unfiltered sample. Both COD_{Colloidal} and COD_{Soluble} were quantified by Hach Lange kits LCK514, using paper filtered (black ribbon paper filter (Schleicher & Schuell)) and membrane filtered (0.45 μm membrane Cronus filter PTFE) samples, respectively. TOC and its derivatives IC and TC were determined by the Shimadzu TOC analyzer out of paper filtered sample. Concentration of VFA, NH₄⁺ and anions was measured by ion chromatography (Metrohm 761 Compact), using membrane filtered sample. Inductively coupled plasma - optical emission spectrometry (Perkin Elmer Optima 5300 DV ICP-OES) was used to determine the total and soluble elements in unfiltered and membrane filtered samples, respectively. Unfiltered samples were submitted to HNO₃ digestion using microwave induced heating (MWD Milestone) at 148 °C during 45 min prior to ICP-OES analysis. The biogas composition was measured by gas chromatography (Varian CP4900 Micro GC with two separate column models Mol Sieve 5 Å PLOT (MS5) and PoraPLOT U (PPU)).

2.3. Granule separation and characterization

Sludge bed samples of 55 ml were taken at 5, 10, 20 and 30 cm high from the bottom in both reactors. The samples were taken

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