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Nutrients recovery from aquaculture waste for use as fertilizer in soilless growth systems

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Abstract

In coming decades agriculture must produce more food with less space and resources. To overcome these challenges, new farming approaches are needed. Investments in smart soilless production systems may contribute to solve these problems. However, todays hydroponic systems rely on inorganic fertilizers which are mined from scarce and non-renewable resources. The aim of this work was to convert aquaculture waste to valuable fertilizer by aerobic digestion (AD). A mixture of drain-water from swirl separators and backwash-water from particle-filters were collected from a local land-based fish-farm with recirculation of water. Three sets of experiments were conducted in duplicate in batch aerated reactors for three weeks. For the first and second set, pH was kept at 7 and 5, respectively. For the third set, the pH was not adjusted and was in the range of 8-9. Aerobic digestion at pH 7 and 5 showed similar mobilization performance with an increase of soluble macronutrients and micronutrients to concentrations close to or exceeding mineral levels recommended for soilless growth systems. At pH 7, the concentrations of soluble phosphorus (P), nitrogen (N), potassium (K), calcium (Ca) and magnesium (Mg) increased by factors of 12.9, 4.9, 1.7, 3.2 and 2.0, respectively. At pH 5, the same concentrations increased by factors of 19.0, 5.4, 1.5, 5.1, 2.4, and 2.0, respectively. The mineral analysis suggested that the resulting nutrient-rich solution can be used for soilless agriculture. However, the ratio of NO_3-N to NH_4-N was low compared to recommended values. Further studies will be carried out to stimulate nitrification during AD to produce a better balanced

nutrient solution and to evaluate the performance of the recovered nutrient solution on plant growth.

Keywords: aerobic digestion, aquaponics, hydroponics, mobilization, nutrient solution, nutrient recovery, organic fertilizer

Introduction

By 2050 population growth will increase to reach 9 billion people (FAO, 2016). More food will be required. At the same time, arable farmland is decreasing because of urbanization and soil contamination (Kozai and Niu, 2016). Therefore, to provide the required food for the future population with less space, more investment should be made in soilless growth system. Nutrient solution which is composed of water and fertilizer is a determining factor in soilless growth systems. However, fertilizers are mined from scarce and non-renewable resources. It has been reported that nutrient depletion is currently a major problem (Kupfernagel et al., 2017). Phosphorus for example may be depleted from apatite mines within 150 years (Mehta et al., 2015). Other sources of nutrients for soilless growth system urge to be found.

In land-based aquaculture, wastewater and sludge from treatment processes contains nutrients in high amount. Approximately 70% of the phosphorus (P) and nitrogen (N) fed to fish can be found in the wastewater. The majority of P in the wastewater is associated with solids, while for N, only 15-20% is associated with solids and the remainder is dissolved (Del Campo et al., 2010). Nutrients in sludge collected from land-based aquaculture exist in three different forms: ionic or soluble form which is the only form assimilated by plants, organically associated nutrient and inorganically bound nutrient (e.g. iron phosphate). These two last forms are not readily available for plant growth. To be utilized for fertilizing purposes, the nutrients associated with particulate compounds should be mobilized into soluble forms. It has been shown that anaerobic treatment of aquaculture sludge from an aquaponic facility allowed an increase in soluble N, while soluble P remained unchanged (Monsees et al., 2017). The objective of the current study was to assess the potential of aerobic digestion (AD) to mobilize nutrients from aquacultural waste at various pH-values for subsequent usage of the recovered nutrients in soilless growth systems.

Materials and methods

Sludge-water consisting of a mixture of drain-water from swirl separators and backwash-water from particle-filters was collected from a recirculating aquaponic system located at the Norwegian Institute of Bioeconomy Research (NIBIO), Landvik, Norway. The sludge-water was distributed to six 25-L polyethylene reactors covered with a lid to prevent evaporation and equipped with diffusors to provide aeration at a rate of 20 L min^{-1} per reactor. Three sets of batch experiments were conducted in duplicate for three weeks. For the first and second set, pH was kept at 7 and 5, respectively. For the third set, the pH was not adjusted and was in the range of 8-9. The pH-values were measured daily with a calibrated pH-meter and adjusted with a diluted HCl-solution to maintain the target pH-values. Samples were collected weekly and analyzed for total suspended solids (TSS), volatile suspended solids (VSS), soluble chemical oxygen demand (SCOD), total phosphorus (TP), total nitrogen (TN) and soluble nutrients. TSS and VSS were determined according to standard method using a pre-weighed 1.6 µm Whatman glass fiber filter. TP was measured according to the NS EN ISO 1568-2 method and TN was determined according to NS 4743 method by the Eurofins laboratory, Norway. Soluble nutrient analysis for ammonium (NH_4^+-N), nitrate (NO_3^--N) and nitrite (NO_2^--N) was performed on a QuAAtro continuous segmented flow autoanalyzer while the other soluble nutrients concentrations were measured by inductively coupled plasma optical emission spectrometry (ICP-OES) by the LMI laboratory, Sweden. All samples for soluble nutrients analysis were pre-filtered through 0.45 µm filters.

Statistics

The aerobic digestion experiments were conducted in duplicate. Mean values with standard deviations are presented. Where standard deviation bars are not observable on the graphs, they do not extend beyond the dimensions of the symbols. Data comparison was done using the statistical t-test with a statistical significance level of $p<0.01$.

Results and discussion

Characterization of the aquaculture wastewater

The concentrations of TSS, VSS, total COD, TP, TN, SCOD and nutrients of the untreated sludge-water are shown in Table 1. As indicated, the sludge-water was a dilute waste stream, with a TSS content of 0.87%. The majority of the solid was organic compounds (VSS), indicating that biological aerobic treatment will degrade organic matter and simultaneously release nutrients from the biosolids. Most of the organic compounds in the sludge-water were associated with particulate matter, with only 2.5% SCOD of the total COD. High degree of particulate association was also the case for N and P. Of the dissolved N-compounds, NH_4^+-N was the only detectable compound, comprising 26% of the TN. Fish excrete dissolved N as ammonia via the gills, and as urine (urea), which is easily hydrolysed to ammonia. As also shown in Table 1, the percentage of dissolved P was low, only 6% of TP.

Effect of aerobic digestion on nutrient mobilization at various pH-values

During the 3-week aerobic digestion period, all dissolved macronutrients increased significantly at pH 7 and 5, while no increases were observed for soluble N, Ca and Mg at unadjusted pH (Figure 1). At pH 7, the concentrations of soluble P, N, K, Ca, Mg, and sulfur (S) increased by factors of 12.9, 4.9, 1.7, 3.2, 2.0, and 2.7, respectively, and at pH 5 by factors of 19.0, 5.4, 1.5, 5.1, 2.4, and 2.0, respectively (Figure 1). By using the t-test with a statistical significance level of $p<0.01$, the differences in solubilization between pH 7 and pH 5 were significant for P, K, Ca, S, Zn, Cu, Fe and Mn. For the remaining nutrients, differences were statistically insignificant. The concentrations of dissolved N, P and Ca after 3 weeks of AD at pH 7 and 5 exceeded the recommended levels of these nutrients in hydroponic solutions for soilless growth system (Mattson and Peters, 2017) by factors from 5 to 9 (Table 2).

Table 1. Characteristics of the aquaculture sludge-water before aerobic digestion.

Parameter	Mean	Standard deviation
pH	7.9	0.01
SCOD (mg L^{-1})	147	5
COD (mg L-1)	6000	800
TSS (g L^{-1})	8.7	0.1
VSS (g L^{-1})	8.55	0.35
Total P (TP) (mg L^{-1})[1]	135	5
Total N (TN) (mg L^{-1})[1]	705	15
B (mg L^{-1})	0.14	0.01
Ca (mg L^{-1})	53.85	0.75
Cu (mg L^{-1})	0.03	0.01
K (mg L^{-1})	62.5	0.5
Mg (mg L^{-1})	21.9	0.2
Mn (mg L^{-1})	0.24	0.01
Mo (mg L^{-1})	0.01	0.003
NH_4-N (mg L^{-1})	181	2
NO_2-N (mg L^{-1})	0.03	0
NO_3-N (mg L^{-1})	0.1	0
P (mg L^{-1})	8.2	0.25
S (mg L^{-1})	26	1
Zn (mg L^{-1})	1.65	0.05

[a]TP and TN include particulate and soluble P and N compounds

Potassium was the macronutrient least solubilized by the aerobic treatment. The observed increases in soluble K by 76% (to 110 mg L^{-1}), 50% (to 94 mg L^{-1}) and 92% (to 120 mg L^{-1}) at pH 7, pH 5 and unadjusted pH, respectively, did not raise the levels above the recommended value of 132 mg L^{-1} (Table 2). At pH 7, all micronutrients rose substantially, except for Mn. In spite of lack of increase, soluble Mn was already at the concentration required for soilless growth systems (Table 2). Half of the dissolved micronutrients (Cu, Mo, Zn) did not increase at pH 5 and remained slightly below the recommended levels. Similar results were obtained for the unadjusted pH.

Nitrogen mobilization

The responses in the different nitrogen compounds under aerobic degradation at various pH values are shown in Figure 2. The TN concentration of unfiltered sludge-water (particulate N + dissolved N) experienced an increase during AD at pH 7 and 5 (Figure 2a, b). Some of this increase can be explained by a 10-15% volume decrease due to evaporation during the 3-week aeration period, even though the reactors were covered. By aerobic degradation at pH 7 and 5 (Figure2 a, b), the soluble nitrogen

(NH_4^+-N + NO_3-N) increased sharply during the first week. After that point, the rate of solubilization levelled off. At the end of the 3-week period, the soluble nitrogen values reached more than 90% of the average total nitrogen concentrations of the samples. The degree of nitrogen mobilization was similar at pH 7 and pH 5, with final concentrations of 890 mg L^{-1} and 885 mg L^{-1}, respectively, which corresponds to 4.9-fold and 5.4-fold increases, respectively. The present finding is much higher than in earlier investigations which reported an increase of soluble N by a factor of 2.56 after aerobic treatment at neutral pH (Delaide et al., 2018), while Monsees et al. (2017) observed a decrease in soluble N from 1 to 0.1 mg L^{-1} after AD at pH 5.

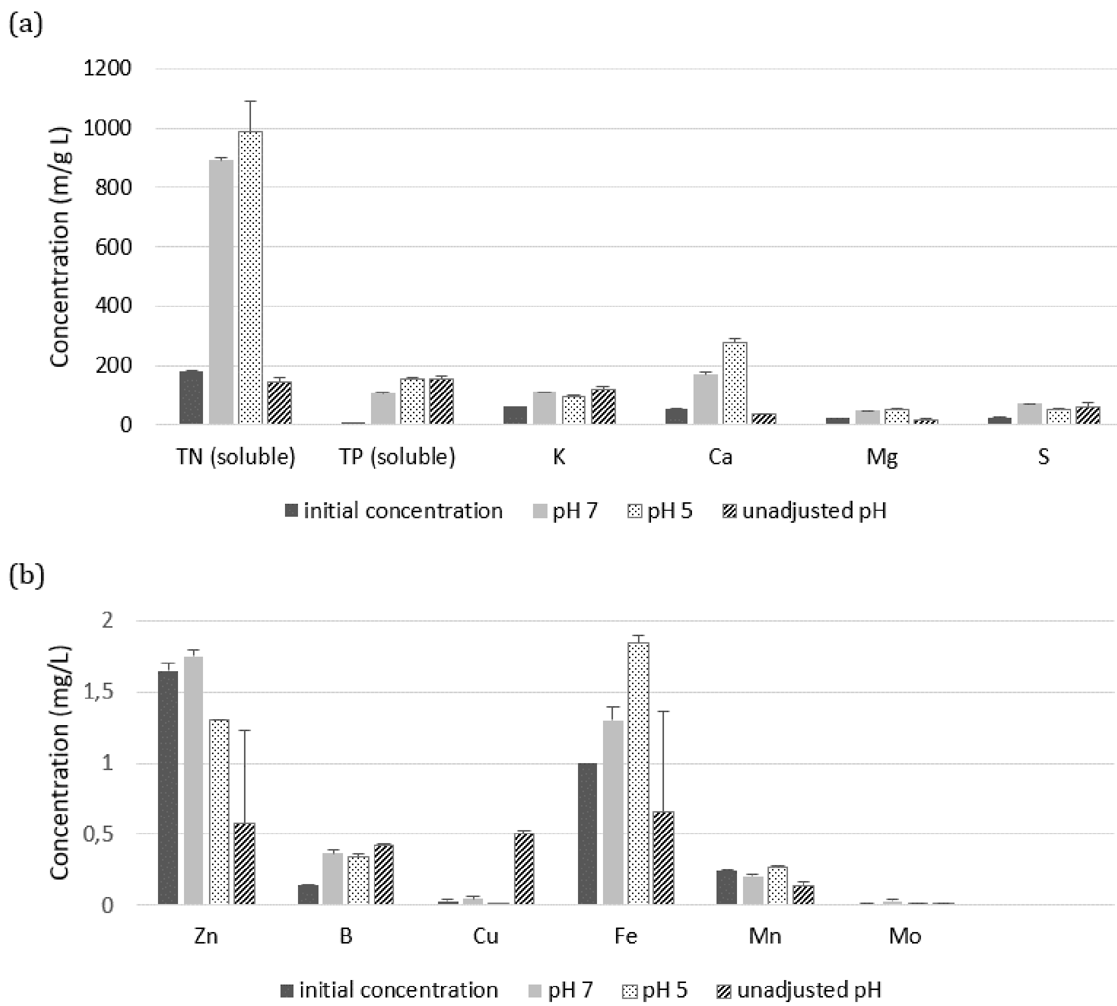

Figure 1. Soluble concentration of (a) macronutrients and (b) micronutrients in the clear-phase after 3 week of aerobic digestion.

Table 2. Soluble nutrients content in the clear-phase of the aquaculture sludge-water after 3 week of aerobic digestion at pH 7, pH 5 and uncontrolled pH, compared to a standard formulation for soilless growth system (Mattson and Peters, 2017).

	pH 7	pH 5	Uncontrolled pH	Recommended concentrations in a standard formulation for soilless growth
NH_4^+-N (mg L^{-1})[1]	862.5	987.5	146	14
NO_3^--N (mg L^{-1})[1]	27	0.2	0.1	160
P (mg L^{-1})	105	155	34	16
K (mg L^{-1})	110	94	120	132
Ca (mg L^{-1})	170	275	35.25	38
Mg (mg L^{-1})	44.5	53	19	14
S (mg L^{-1})	70.5	53	59	-
Zn (mg L^{-1})	1.8	1.3	1.3	0.1
B (mg L^{-1})	0.37	0.34	0.12	0.16
Cu (mg L^{-1})	0.05	0.006	0.03	0.11
Fe (mg L^{-1})	1.3	1.9	1.7	1
Mn (mg L^{-1})	0.2	0.2	0.2	0.2
Mo (mg L^{-1})	0.003	0.001	0.001	0.002

[a]The sum concentration of soluble NH_4^+-N and NO_3^--N was higher than the initial TN. This was partly explained by an increase in TN attributed to a volume decrease of 10-15% due to evaporation.

For AD without pH control, the total N (TN) concentration was reduced from 705 to 225 mg L^{-1} (Figure 2c). This sharp depletion of TN can be caused by dissolution of bound nitrogen to NH_4^+, with subsequent escape of NH_3-gas. At high pH-values of 8-9 as experienced in these reactors, the equilibrium between NH_4^+ and NH_3 will be shifted toward the gaseous NH_3, which may escape to the atmosphere (Glass and Silverstein, 1998). Another possibility for this reduction is that the released NH_4^+ may precipitate in the form of struvite (magnesium ammonium phosphate) at high pH. Without pH control, a modest increase in soluble N was observed during the first week (Figure 2c). Thereafter, a reduction followed to a level below the initial value of soluble N. Compared to the initial soluble N concentration, the decrease was not statistically significant.

Our data shows that the only dissolved nitrogen compound present at pH 5 and without pH-control was NH_4^+-N. At pH 7, approximately 3% of the dissolved nitrogen was in the form of NO_3^--N. This is reflected in Figure 2, which shows that the curves of soluble N and the curves of NH_4^+-N are very similar at all pH-values. These results agree with the fact that nitrification will proceed better at pH 7 than at higher or lower pH-values. The limited nitrification observed even at pH 7 can be explained by high concentrations of organic compounds which will stimulate the growth of heterotrophic bacteria, thereby depressing the growth and activity of the nitrifying bacteria. The ratio of NO_3^--N to NH_4^+-N of the solution after aerobic digestion was low compared to what is recommended for soilless growth solutions. In future research, we will try to stimulate nitrification during AD to increase the NO_3^--N concentration and lower the NH_4^+-N concentration.

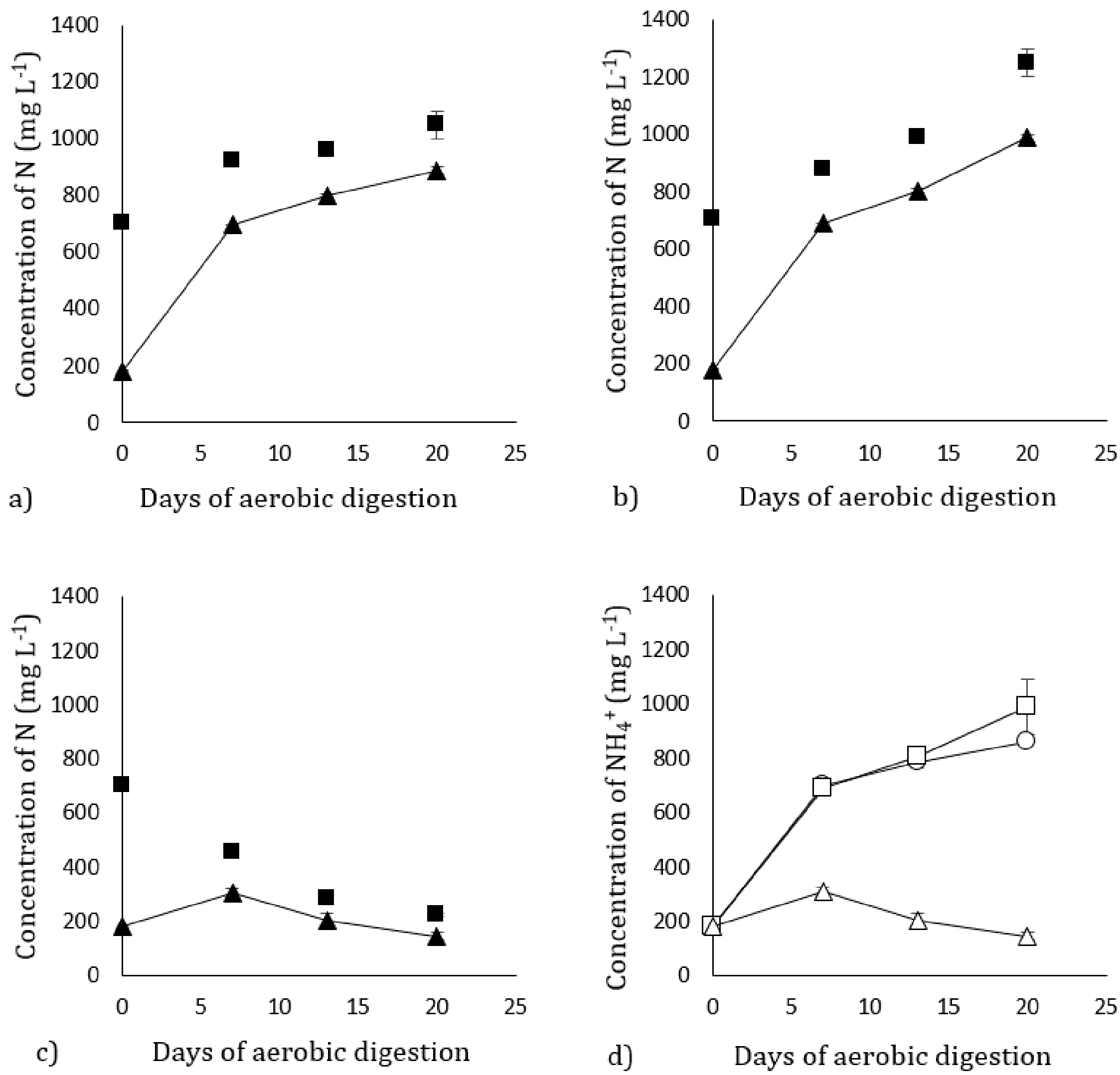

Figure 2. Mobilization of nitrogen during aerobic digestion at (a) pH 7, (b) pH 5, (c) at uncontrolled pH, and (d) NH_4^+-N at pH 7 (○), at pH 5 (□) and at uncontrolled pH (△). ■; total nitrogen (soluble + particulate), ▲; soluble nitrogen (NH_4^+-N + NO_3^--N).

Phosphorus mobilization.

Aerobic degradation of the sludge-water resulted in soluble P increases at all pH-values tested. The highest soluble P concentration was observed at pH 5 with a final concentration of 155 mg L^{-1} which corresponded to a 19-fold increase and to approximately 90% solubilization of the average TP (Figure 3). This degree of solubilization was substantial higher than in earlier published work by Monsees et al. (2017) who reported an increase of soluble P by a factor of 3.2 at pH 5 after aerobic treatment. At neutral pH, the concentration of soluble P was 105 mg L^{-1} at the end of the treatment which corresponded to a 12.9-fold increase and 73% solubilization of the average P. This degree of mobilization was also higher than in earlier published work by Delaide et al. (2018) who reported an increase of soluble P by a factor of 2.18 after AD at neutral pH. Only a modest increase to 34 mg L^{-1} was observed in the reactors without pH-control.

The dynamic of chemical and physical mechanisms related to adsorption/desorption and precipitation/solubilization are altered by pH decrease. Interestingly, the change of concentration of soluble P and Ca^{2+} during AD had similar trend at different pHs (Figure 3e). This is a strong indication that the solubility of P is ascribed to the dissolution of Ca-P compounds.

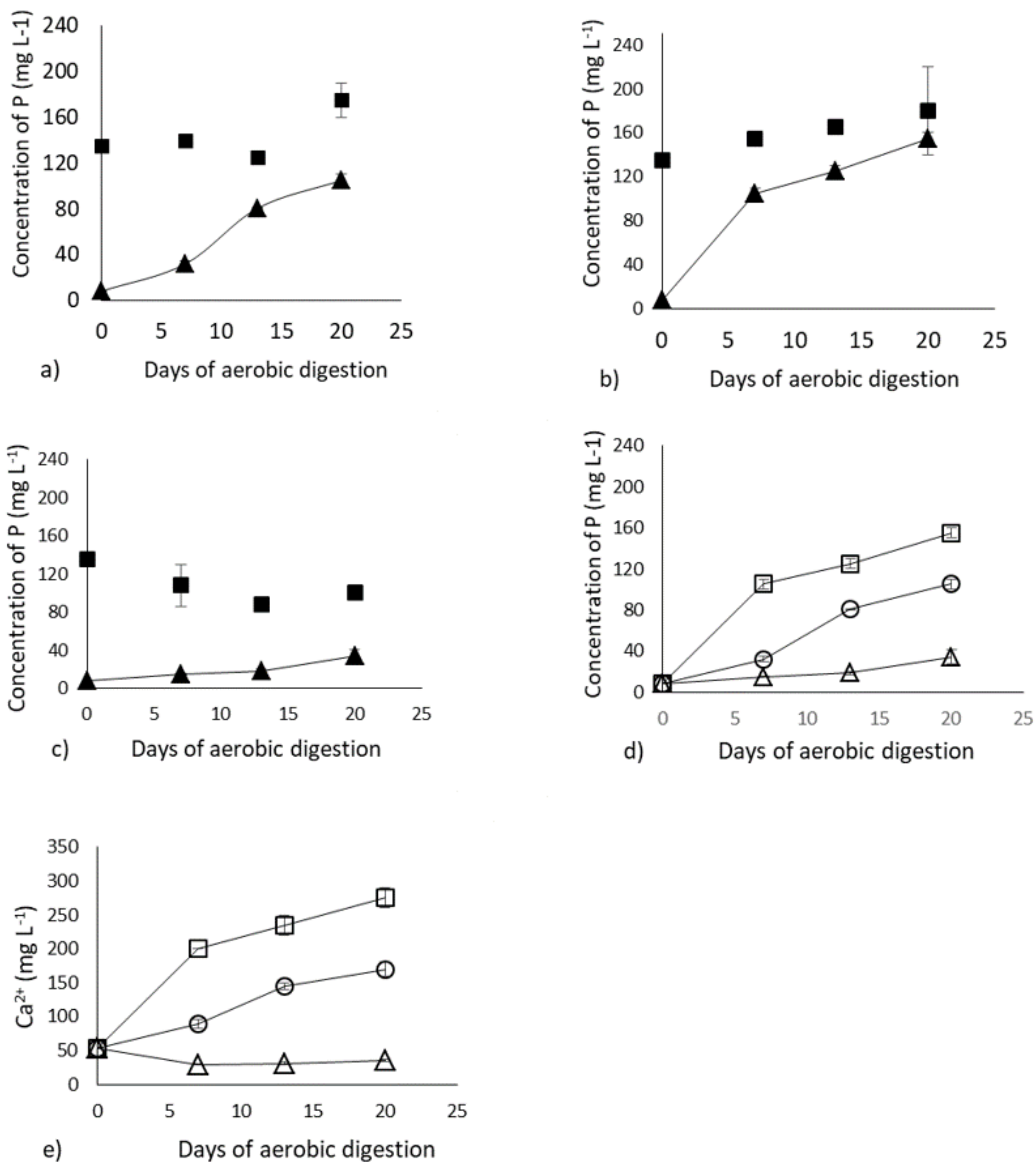

Figure 3. Mobilization of phosphorus during aerobic digestion at (a) pH 7, (b) pH 5 and (c) without pH control and d) concentration of soluble P at pH 7 (○), at pH 5 (□) and at uncontrolled pH (△), (e) soluble calcium (Ca^{2+}) at pH 7 (○), at pH 5 (□) and at uncontrolled pH (△). ■; total phosphorus (soluble + particulate), ▲; soluble phosphorus.

Conclusions

Aerobic digestion at pH 5 and 7 for 3 weeks were both able to mobilize high amounts of nutrients from aquaculture sludge-water. The concentrations of the majority of macronutrients and micronutrients in the digestate after aerobic digestion at pH 7 and 5 were close to or exceed the required mineral levels recommended for soilless growth solutions. The ratios of NO_3-N to NH_4-N of the solutions were low compared to recommended ones for hydroponic growth. Further research will be conducted to stimulate nitrification during aerobic digestion to increase the NO_3-N to NH_4-N ratio, and to verify the performance of the recovered nutrient solution on plant growth in a small-scale hydroponic system.

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