

## **CARBON AND STRUVITE RECOVERY FROM CENTRATE AT A BIOLOGICAL NUTRIENT REMOVAL PLANT**

Harlan Kelly<sup>1</sup>, P.Eng., Dayton & Knight Ltd.,(D&K); Bonita Dirk, E.I.T., D&K; Al Gibb, PhD, P.Eng., D&K; Fred Koch, University of British Columbia (UBC); Don Mavinic, PhD, UBC  
Dayton & Knight Ltd.  
#210-889 Harbourside Drive  
North Vancouver, BC, Canada, V7P 3S1

### **ABSTRACT**

A one-tenth pilot-scale demonstration study of volatile fatty acid (VFA) and soluble phosphorus recovery from an Autothermal Thermophilic Aerobic Digester (ATAD) was carried out at the City of Salmon Arm Water Pollution Control Centre in British Columbia, Canada. VFA recovery returns supplemental organic carbon to the enhanced biological nutrient removal (EBNR) treatment process and simultaneous phosphorus recovery avoids return of phosphorus to the process. The pilot demonstrated that the recovery of VFA and phosphorus was practical and beneficial.

Soluble phosphorus was recovered using a University of British Columbia, (Ostara Nutrient Recovery Technologies) commercialized struvite crystallizer. The crystallizer effluent, which was high in volatile fatty acid (VFA) concentration, was returned to the biological nutrient removal process. Bench-scale batch tests were routinely carried out to observe the impact of the returned VFA on phosphorus removal.

The ATAD process operates at high temperatures (40°C to 70°C) and at the Salmon Arm EBNR treatment plant, the ATAD process solubilizes large amounts of phosphates and ammonia, which results in high nutrient concentrations in the centrate.

The study was successful in showing that:

- Supplemental VFA for the EBNR process could consistently be obtained from a mix of waste biological (phosphorus accumulating organism) sludge and primary sludge,
- Phosphate could be removed from VFA laden sidestream liquors prior to return to the EBNR process, and
- High quality fertilizer struvite pellets (avg. 2 mm diameter) could be consistently obtained by removing approximately 80% of the phosphorus and (although unexpectedly high), 35% of the ammonium-nitrogen from the centrate.

## **KEYWORDS**

Volatile fatty acid (VFA) for EBNR, phosphorus and ammonium recovery, struvite crystallizer, ATAD, thermophilic, nutrient recovery.

## **INTRODUCTION**

The purpose of the demonstration pilot at the Salmon Arm Water Pollution Control Centre (WPCC) is to show that a fermented mix of biological and primary sludges, from an enhanced biological nutrient removal (EBNR) treatment plant, could be a source of volatile fatty acid (VFA) for phosphorus accumulating organisms (PAO) and denitrifiers. The pilot included a full scale acid digester, through use of the WPCC autothermal thermophilic aerobic digester (ATAD), a one-tenth scale struvite crystallizer (from the University of British Columbia and Ostara Nutrient Recovery Technologies Inc.), the plant centrifuge, and two centrate storage tanks.

The use of fermenters to produce VFA for EBNR plants is not new. The method of production however, has been largely restricted to use of primary sludge as reported by Rabinowitz (1985, 1994), Bundergaard (1992), Lötter (1992), Barnard (1994), and Chu (1999) since this was the most likely source of readily available carbon. While others, including Fothergill (1994, 2000), Chu (1992, 1994, 1996), Usisik (2008) and Jiang (2007) have shown the importance of waste biological sludge as a source of VFA, no full scale facilities are known at this time to be making use of this secondary sludge resource. The absence of the use of waste biological sludge may be due to the concern that EBNR biological sludge will release phosphorus into the liquid train and largely limit or nullify the value of the returned VFA. This has possibly influenced EBNR plant designers to separate the solids management into two separate treatment trains, one for the primary sludge and one for the waste biological. Or where all the sludge is biological, the source of VFA from the biological sludge is not considered and a supplement such as methanol or acetate may need to be purchased. With the exception of a few researchers (Rabinowitz (1985) and Elefsiniotis (1994, 2004)), the development of the fermenters has also largely overlooked the large body of knowledge of acid digesters from Ghosh (1974, 1975, 1987, 1995, 1997, and 2003) in favour of hydraulic residence time (HRT) controlled sidestream thickeners and completely mixed stirred tank (CMST) reactors. The development of the current fermenter design appears to have been partly in response to the EBNR researchers such as Lötter (1992), who recognized VFA to be an important substrate for the EBNR process, and the related earlier discovery that gravity thickeners could supply this source when used appropriately, (Oldham (1984), Barnhard (1994)).

This research is to demonstrate that the use of a biological sludge containing PAO is appropriate for VFA recovery and reuse in EBNR treatment, and the use of a sludge retention time (SRT) controlled acid digester is a preferred means of VFA production for both primary and biological sludge. The study is to show that the use of biological sludge is desirable when the phosphate can be recovered as a side benefit. The VFA recovery can be mainstream or sidestream in the acid digester and struvite crystallizer process trains as presented in the research results of the report by Dayton & Knight (2008).

The pilot operated at the crystallizer capacity of 1.7 L/minute and compares to the centrate production of about 18 L/minute.

### **Fermentation Technology for EBNR**

The development of fermentation systems to enhance biological uptake of nutrients appears to have developed primarily in the nutrient removal field of wastewater treatment technology as a side stream process. This has taken shape largely through the introduction of sewage wastes that were observed to have highly soluble COD or low molecular weight volatile fatty acids (VFA), particularly acetic and propionic forms. Where plants did not have sufficient VFA in the incoming sewage, diversion of supernatant from gravity sludge thickeners, and later the development of side stream fermenters/thickeners was shown to provide a needed VFA supplement (Oldham, 1984), which led to advancement in fermenter design. Optimization studies showed continued benefit in enhancement of the nutrient removal process. This has promoted the development of separate fermentation reactors to produce VFA, (Barnard (1983); Rabinowitz (1985 and 1994), Elefsinotis (1994), Oldham (1994)). The conventional process that evolved from the static fermenter/thickener remained in a side stream configuration, but became a relatively simple process that used a completely mixed reactor tank (CMRT) to allow fermentation of primary sludge followed either by sending the fermented product to a downstream dedicated thickener, or returning it to a primary clarifier. Use of primary sludge alone avoided release of phosphorus from the waste biological sludge and its return to the EBNR process. The fermented product was elutriated in the primary or thickener tanks to carry the soluble fermented products to the biological plant. The conventional CMRT fermenter is reported to require about 7 to 15 days of solids retention and low mixing energy of about  $16\text{W/m}^3$ . The fermenters operate at nominal temperatures of the wastewater using sludge retention time control and solids concentration monitoring. Volatile solids concentration ranges from 1000 mg/L to 2000 mg/L. Soluble COD is about 35 to 40% and VFA is about 20 to 35%.

Several key research articles identify a map of recent progress in WWTP production and use of VFA for EBNR facilities. These are summarized and the key findings are noted as a basis for the pilot work.

Ghosh (1987) provided an extensive report on two-phase anaerobic digestion that identified ranges of VFA production from 1500 to 4000 mgVFA/L as acetate and identified an optimum pH of about 5.5 to 6.6 in an acid digester operating at 2.2 days HRT. Lipid and protein reductions were highest in this range, while carbohydrate reduction remained the same.

Ghosh (1995) reported for studies on a full scale 2-phase digestion in DuPage County Ill. The acid digester when operated at between 1.75 to 2.2 days HRT contained VFA concentration from 5950 to 9790 mg/L for VS reductions of 17.1 to 24.3%. This represents VFA generation of between 114 to 174.5 mg VFA/g VS. The optimum acid concentration was obtained at the 1.75 day HRT. Both mesophilic ( $36.8^{\circ}\text{C}$ ) and thermophilic ( $49.8^{\circ}\text{C}$ ) pilot tests were carried out and results showed slightly higher gas generation (higher VFA generation) with the thermophilic digester. The sludge was a mix of waste biological and primary municipal sludge.

Elifsiniotis (1994) reported VFA production from fermentation of municipal primary sludge to range between 167 to 200 mg acetate (HAc)/g VS; (the fermenter operated at a SRT of 10 days and an optimum HRT of 12 hours between 18°C - 22°C). About 44% to 48% of the VFA was reported as acetate and about 30% to 37% was reported as propionate. Liquid and solid phase separation was used to optimize HRT while using a long sludge age provides greater opportunity for fermentation and digestion. The longer sludge age allowed methanogens to convert VFA to gas. The authors note that the 12 hour optimum did in fact produce gas, and longer HRT's further increased the gas production. The 10 day sludge age requires a larger reactor to contain the sludge. The solids were thickened to about 1% to 2% VSS from a 3g/L VS feed.

In later work, Elifsiniotis (2004) showed that acetate was the preferred VFA for denitrification and showed that VFA in general was preferred over other carbon forms.

Chu (1994) reported on several full scale fermenters. Production of the Penticton fermenter was 28 mg/g TS for a 12.6 day SRT, and the Kelowna fermenter was 12.6 mg/g TS for 18.8 day SRT. Both operated between 10°C to 24°C. The VFA of these fermenters comprised on average about 43% to 48% acetate. The balance is largely propionate. The VFA generated at these plants was a fermentation product of primary sludge alone. Chu's work however, showed that when using micro-aerobic thermophilic digestion (TAD) on primary sludge, for a 3 day SRT operating at 37 to 46°C, VFA production was 56 mg VFA/g TS and the VFA was 81% acetate. In a comparative test of the Salmon Arm ATAD reactors, VFA production was shown to be about 39 mg VFA/g TS in a 5 day SRT at 35°C – 50°C and the VFA was 70% acetate. The sludge at Salmon Arm is a mix of primary and waste biological sludge from a biological nutrient removal treatment process; the ATAD and EBNR processes are described by Kelly (1993 and 2005). (For the ATAD and the TAD investigation, SRT is the same as HRT.)

Fothergill (1996 and 2000) reported investigations of the fermentation of primary sludge and waste biological in differing ratios from the University of British Columbia pilot EBNR wastewater treatment plant. The fermentation was undertaken in a 7 day HRT thermophilic aerobic digester. Operation at an Oxidation Reduction Potential between -300 to -400 mV showed that for 13.9 g/L VS feed, VFAs were produced ranging from 100 to 1230 mg/L (average 490 mg/L) and phosphate as P was released at an average concentration of 118.7 mg/L. The VFA produced was about 35 mg VFA/g VS. VS destruction was reported as 30.4% and the VFA produced is calculated at 116 mg/g VS destroyed. The phosphate VFA:PO<sub>4</sub>-P ratio was 4.1. This suggests that an insufficient amount of VFA would be produced in the tested sludge to adequately remove the phosphate if a ratio of about 8:1 VFA:PO<sub>4</sub>-P is required. For this case, either a greater VFA mass must be produced or the phosphorus must be separately removed by chemicals.

Wang (1997), has shown that high temperature hydrolysis of waste biological sludge at 60°C and 80°C will increase gas production of anaerobic digesters by over 30% through introducing VFA (as measured by COD) of over 2000 mg/L untreated (control), 5000 mg/L at 60°C and 5500 mg/L at 80°C.

Cheunbarn (1999) undertook bench scale laboratory work to determine an optimum aerobic thermophilic digestion HRT to achieve pretreatment to maximize subsequent anaerobic digestion treatment. (The HRT and SRT were equal in the complete mix bench scale reactor.) Thickened

waste biological and primary sludge (1:1) from a municipal treatment plant was used as feed. Total solids feed ranged from 3 to 5%. Average SRTs for the study were 0.6, 1 and 1.5 days and temperatures ranged from 55°C to 65°C. Average feed VS was 28.5 g/L, average COD was 20 to 22 g/L, and average VFA as acetate (HAc) was 960 mg/L. The average effluent VFA concentrations from the reactors were 1900 mg/L at 0.6 d SRT, 2200 mg/L at 1.0 d SRT and 2500 mg/L at 1.5 days SRT. VS reduction was 35-42%, and COD reduction was about 33%. VFA production is estimated to be 67 to 88 mg VFA/g VS, or net VFA production is estimated at about 100 to 125 mg/g VS destroyed. The VFA characterization was not reported.

Operation staff at the Salmon Arm WPCCC has used chemicals for removal of the solubilized phosphorus, as noted by Kelly (2000) since 1999, and the centrate containing VFA has been returned to the primary tanks to add to the VFA supplement for the EBNR process since 1998. The plant has operated successfully with 50% effluent concentration of ortho-phosphate-P of less than 0.07 mg/L and 90% less than 0.11 mg/L. The recovery and return of VFA to the Salmon Arm EBNR plant appears to provide consistency in the performance of biological phosphorus removal.

Puchajda (2005) undertook bench scale testing to determine a maximum acidification (hydrolysis) rate that was shown to take place in the first day and then dropped in 2<sup>nd</sup> to the 5<sup>th</sup> day. An optimum rate at about 3 days showed a VFA increase from 2500 to 6500 mg/L when fermenting a mix of primary sludge and waste biological sludge at 55°C. The COD feed was 10.4 g/L, which suggests a VS of about 8 g/L or less than 1%. The VFA production at this concentration would be about 550 mgVFA/g VS. This is compelling if the data are correctly interpreted by these authors and supports work reported by Kelly (1990).

In a study reported by Jiang (2007), VFA production from fermentation of waste biological sludge was shown to be enhanced in the presence of a natural surfactant (sodium dodecylbenzene sulfonate). An optimum dose of 20 mg surfactant /g (dry sludge) was found. Maximum concentrations, obtained in batch reactors at 20°C were about 2600 mg COD/L at 6 days fermentation, 3285 mg COD/L at 12 days and 2675 mg COD/L at 15 days, compared to the blank test of 339 mg COD/L at 6 days. Another feature of the report identified the effect of struvite formation on the soluble COD concentration. When struvite formation was undertaken, 93% of the soluble phosphate and 22% of the ammonium were removed. Soluble COD was reduced by 4% during the struvite formation.

Ucisik (2008) reported VFA production from fermentation bench scale tests on primary, waste biological and mixed sludges from 6 Danish municipal wastewater treatment plants. The fermenters included both continuous CSRTs operated at 20°C for 5 day HRT, and 6 identical semi-continuous reactors operated at 37°C for 5 day HRT. For the CSRT fermenters, the net VFA production for five sludge sources varied from 11.3 to 25 mg COD/g VSS. For the semi-continuous flow reactors, the net VFA production was highest for primary sludge as 168 to 197 mg COD/g VSS and lowest for waste biological as 62 mg COD/g VSS. Mixed sludge gave an average of 114 mg COD/g VSS. With the exception of one sludge source that gave a lower percentage of acetate relative to propionate, the acetate generally was highest varying from 40 to 80% of the VFA. Release of ammonium and phosphate in the liquid fraction was shown to vary from about 1 to 5 mg NH<sub>4</sub>-N/g VSS and 1.1 to 5.7 mg PO<sub>4</sub>-P/g VSS. For a ratio of 8:1 (VFA: PO<sub>4</sub>-P) to ensure

adequate VFA for P removal, the VFA would need to be about 10 to 50 mg/L to offset the observed phosphorus release for the fermented biological sludge.

Farrant (2008) reported VFA production from sludge fermentation of about 660 to 1000 mg VFA-COD/g TSS. The investigation shows that the use of the sludge for producing VFA has excellent response in PAO anaerobic phosphate release and aerobic phosphate uptake from the soluble fraction. The work suggests that further research is needed to examine long term response. In a related report by Le (2008), study findings of VFA production at 43°C gave a product of about 30 mg VFA/g VS and the highest production rate at an HRT of 12 to 18 hours. The fermenter operates as a non-mixed phase separation device using flotation from natural gas production to float the solids above the fermented liquor. The phase separation showed increased performance efficiencies as temperatures increased. VFA characterization was not given.

An acid digester forms the first cell of a series of reactors of an Autothermal Thermophilic Aerobic Digester, (ATAD). Prior to 1988, full scale use of the high temperature aerobic digestion process was not yet introduced to North America and was being used in Europe only, (specifically the United Kingdom and Germany). In 1986 through 1988, three ATAD plants were designed by Dayton & Knight Ltd. and constructed at three British Columbia municipalities. These facilities were the subject of a one year demonstration study. The nature of the investigation is reported in numerous papers prepared by Kelly (1989 through 2003) and Layden (2007).

The reviewed literature and related past experience illustrate the following:

1. VFA may be produced from primary sludge in greater amounts than from waste biological sludge at low temperatures where little hydrolysis of the biological mass is shown to occur,
2. Enhanced hydrolysis of the biological sludge is shown to occur during high temperature fermentation, in alkaline conditions and where natural surfactants are present,
3. VFA produced from acid digester processes appeared to provide a valuable substrate for the EBNR process and does not inhibit but improves the stability of phosphorus removal and denitrification,
4. Waste biological sludge can be a valuable source of VFA and can supplement the production of VFA normally obtained from primary sludge alone; a means of removing soluble phosphate and ammonia improves the benefit of the fermentation of waste biological sludge.
5. Micro-aerophilic ATAD and TAD processes are shown to produce a higher concentration of the preferred VFA as acetate over propionate and butyrate, for EBNR.
6. Use of chemicals for soluble phosphate removal or the use of a struvite crystallizer to remove both ammonia and soluble phosphate, does not appear to reduce the available VFA for use in an EBNR process; the struvite crystallizer may however, be impeded by high concentrations of TS in the feed,
7. Micro-aerophilic thermophilic processes such as TAD and ATAD will minimize methane formation while providing high concentrations of VFA in short fermentation times of 1 to 5 days,
8. A probable optimum HRT and SRT is about 1 to 2 days for maximizing the VFA in an acid digester, TAD or ATAD. In the latter case it would mean using the first stage of a series digester and loading it to a sufficiently high rate to achieve the desired SRT.
9. Probable pH range for optimum production of VFA is 5.5 to 6.

## METHOD

The ATAD reactors were used for the VFA production. The feed to the ATAD reactors is a mix of EBNR waste biological sludge and primary sludge at a 30:70 ratio. The primary feed averages 4.5% TS and is 84.5% VS. The waste biological sludge (WBS) is thickened to 5.2% TS and averages 86.1% VS. The combination of sludge feed was 5% TS of which 84.1% was VS. Combined sludge loading to the first cell of the reactor is 20.2 kg TS/m<sup>3</sup>-d and 17.2 kg VS/m<sup>3</sup>-d. The ATAD consists of 5 cells; the first three are in series and the last two small cells are in parallel-used as post ATAD or storage cells prior to centrifuge dewatering. In the 11.7 day average SRT, the reactor provides a 42.2% TS and a 49.6% VS destruction. Evaporation increases the SRT by about 0.8 days, which is included in the 11.7 days.

Table 1 shows the VFA profile through the reactors prior to the pilot operation. The VFA is seen to decrease through the reactors but increase in the final reactor possibly due to the lower aeration provided and the constant level changes that result from the use of the centrifuge.

**Table 1 - VFA Concentration in ATAD Cells**

Sample	Total VFA Conc. as Acetic Acid (mg/L) Parsons(2008)	HRT (days)		Temperature °C
		July (2008)	August (2008)	Average July August (2008)
First (Feed)	2332	2.4		37
Second	742	3.4		62
Third	112	3.0		63
Fourth (Final)	867	2.1		55

In a study of the Salmon Arm ATAD by Parsons (2008), the protein content of the mixed feed sludge was found to vary between 1.2 to 1.5 g/100 g of TS. Carbohydrate varied between 1.2 to 2.5 g/100 g TS and fat content varied between 0.75 to 0.9 g/100 g TS. This study identified that oxidation reduction potential was below -400 mV for all reactors, and pH varied from 7 to 8.2 for HRTs of 7 to 20 days.

The ATAD reactors release soluble phosphorus and produce ammonia in high concentrations.

The proprietary struvite crystallizer, developed at the University of British Columbia and commercialized by Ostara Nutrient Recovery Technologies, was used to remove phosphate in crystal form from the dewatering centrate of the plant. This process is described by Britton (2005).

The crystallizer was operated using ATAD centrate because of the relatively high concentration of VFAs in the fourth cell and the advantage of eliminating a previously planned solids-liquid separation step that would have had to be used from a diversion from the first cell. Also it was

necessary to minimize the disruption to the plant operation and maintain the Class A product so short circuiting to the fourth cells was not considered.

Normally, alum is added to the liquid before centrifuging to retain the phosphorus in the solid fraction. For the crystallizer to function, the phosphorus must remain in solution in order to enable reaction with the magnesium and ammonium to form pellets in the crystallizer. Polymer is also normally added to the liquid in order to coagulate the solids for easier separation. The centrate used in the crystallizer was given a higher dose of polymer than normally used and no alum.

The struvite crystallizer was used to capture the phosphorus and ammonia, and “batch tests” were used to simulate the effect of returning the effluent to the treatment process. During the study, dissolved ortho-phosphate, dissolved magnesium, Volatile Fatty Acid (VFA) content, pH, temperature, conductivity, ammonium, and Total Suspended Solids (TSS) were monitored. To assess the performance of the crystallizer, magnesium, ammonium, phosphate, pH, conductivity, temperature, solids, and VFA concentration were measured daily. An on-site a method to measure Mg was developed and optimized, (Dayton & Knight Ltd. 2008).

The crystallizer is a fluidized bed reactor, with sections of increasing diameter and a settling zone on top. The changes in diameter result in turbulent eddies at each transition and ensure sufficient mixing in the reactor. These different sized sections also help to classify the fluidized particles by size, so that only the largest ones will be harvested (Britton et al., 2005). From the top, the sections have diameters of 77 mm, 52 mm, and 40 mm respectively. Below this is the harvest zone. Figure 1 shows the changes in diameter and the size of the reactor. This pilot reactor treated 1.7 L/min of centrate which is approximately 10% of the overall centrate (18 L/min).

**Figure 1: Crystallizer Assembly**



During the centrifuge dewatering, the centrate was stored in two equalization tanks. Figure 2 shows the cylindrical tank with the conical bottom that was used for the study. Each time the tanks were filled the centrate was allowed to separate and solids settle out overnight or longer in both tanks, before the supernatant was used in the crystallizer. The following day, the biosolids were drained from the bottom of the tanks, and before the tanks were refilled, the tanks were emptied of any sludge or foam. The supernatant, (clarified centrate) was pumped from the cone shaped tank up through the bottom of the crystallizer. The overflow from the crystallizer went into the crystallizer clarifier, and the overflow from the clarifier was recycled back into the crystallizer at multiples of the influent flow rate.

**Figure 2 - Cylindrical Centrate storage tanks with conical bottom (in lower floor), taken during assembly of Crystallizer**



A process flow diagram of the pilot plant set-up and all included tanks and pumps is shown in Figure 3.

### **Volatile Fatty Acids (VFAs)**

Samples for VFA analysis were taken five days a week and preserved for analysis by gas chromatography, using phosphoric acid to reduce the pH below 2. The VFA samples were analyzed at the UBC Civil Engineering Department Environmental Lab by a Gas Chromatograph using an HP-FFAP column as described in Hewlett Packard Application Note 228-398, January 1998. The separation of unsaturated acids and fatty acid methyl esters was undertaken using HP-FFAP and HP-INNOWax Columns.

## Phosphorus

Dissolved ortho-phosphate was measured as phosphorus, both in the influent and the effluent of the crystallizer, approximately five days a week. The samples were filtered through 1.2  $\mu\text{m}$  filter paper, then suctioned through 0.45  $\mu\text{m}$  filter paper and analyzed using a spectrophotometer according to *Standard Methods* Method 4500-P D. However, because the concentration of  $\text{PO}_4\text{-P}$  is consistently very high, the samples must be diluted significantly to bring them into the range of the spectrophotometer. Therefore, only 0.1mL samples are used for each test and this can introduce some error.

## Magnesium

In order to measure the magnesium concentration in the centrate on a daily basis, the Calculation Method is used from *Standard Methods*, Method 3500-Mg E except that polyaluminum chloride (PAC) was added to remove soluble phosphate, which is known to interfere with the test. In order to ensure all phosphates had been removed, an alternative method was developed by adding small amounts of PAC to the samples, centrifuging them and pouring off the centrate. Then another smaller volume of PAC would be added to the centrate and the samples centrifuged again. Finally, this was done a third time and the  $\text{PO}_4\text{-P}$  measured in the centrate. This centrate was then used for the hardness tests. Using this method, the  $\text{PO}_4\text{-P}$  was always zero in the centrate or below the concentration at which it is known to interfere with the test.

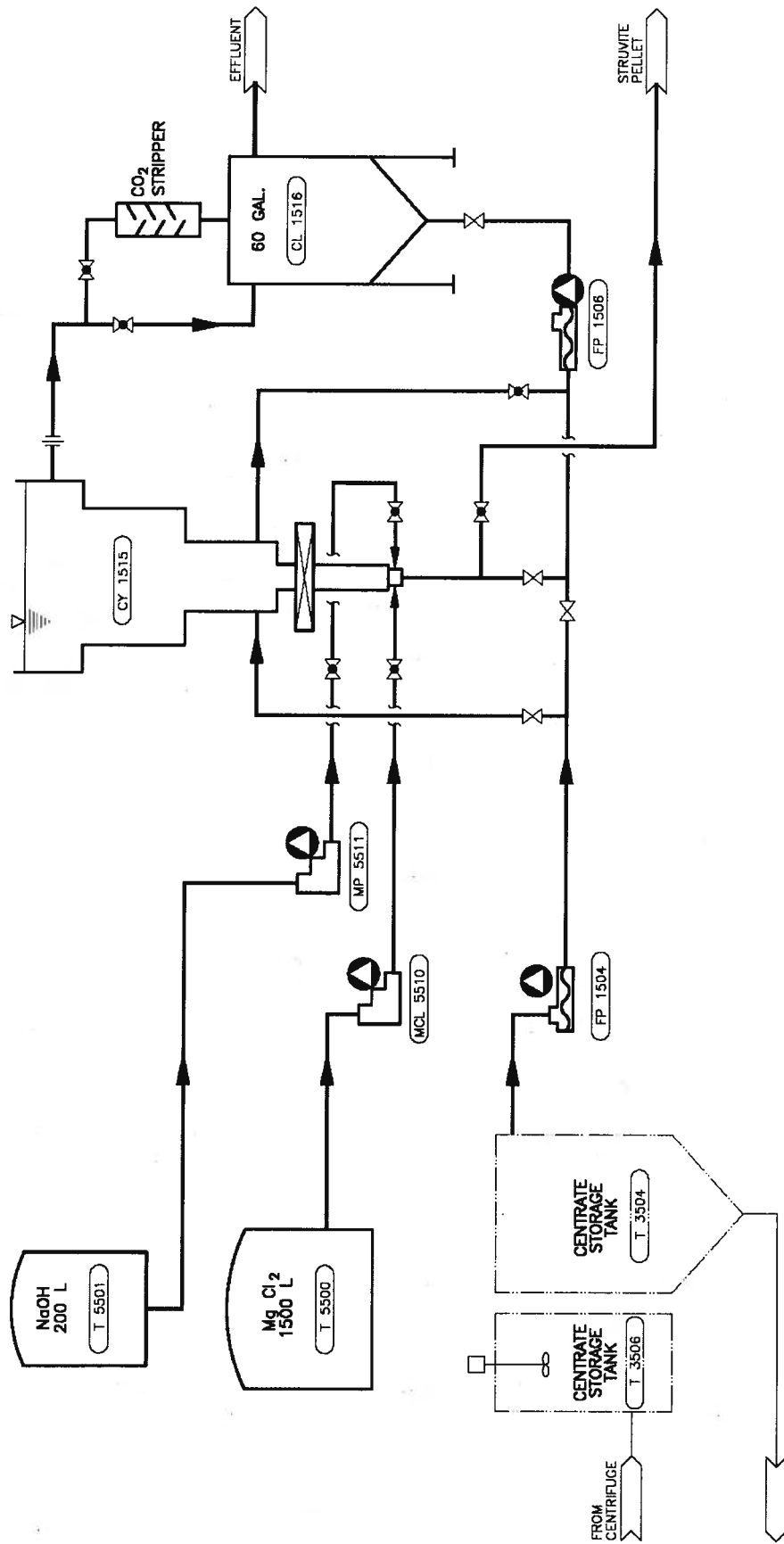
Because of the high level of solids in the centrate, this test was confirmed by also sending samples for analysis by atomic absorption to UBC Department of Civil Engineering Environmental Lab. There, the magnesium was analyzed according to *Standard Methods*, 3111 B using the Direct Air-Acetylene Flame Method. The magnesium samples were prepared for analysis using *Standard Methods* 3030 E. Nitric Acid Digestion method. The final method developed at the lab gave the most accurate results.

## pH

Because of its influence on phosphorus solubility, pH is a key parameter. If the phosphorus is not in solution, then it is not available to bind with the magnesium and ammonium to form crystals. Because of the importance of pH, an in-line pH probe is used to regulate the supply of caustic to the reactor, so that the desired pH is achieved. This probe is located at the top of the harvest zone (the bottom section of the reactor). A second pH meter measures the pH in the clarifier, from which flow is recycled back into the crystallizer. This second meter is used to confirm the readings given by the in-line meter.

The key parameter for optimal operation of the crystallizer is the super saturation ratio (SSR) (Rahaman, 2008). Below a saturation ratio of one, no crystals will form, as the solution is not saturated. Based on an optimal value of the SSR, the flow is determined. The flow rate of influent centrate for this reactor was approximately 1.7 L/minute and stayed constant for the length of the study. This compares with the overall centrate rate of 18 L/minute. After the model in-puts are updated each day, the model calculates the optimal pH, and the controller set point is adjusted based on this.

Figure 3 - Process Flow Diagram



## **Ammonium**

Dissolved ammonium concentration was determined using the Ammonia-Selective Electrode Method (Method 4500-NH<sub>3</sub> F from *Standard Methods*). This test converts all the dissolved ammonia and ammonium to ammonia by raising the pH above 11. Using this test together with the measured pH of the influent the dissolved ammonium-nitrogen concentration can be calculated. This pH measurement was taken using a hand-held pH probe.

This test was done five times per week and the results were entered into the model.

Some tests were also done at the UBC Environmental Engineering Lab, to confirm the results obtained on-site. These tests were done using the phenate method on the Lachat QuikChem 8000 flow injection analyzer. On-site measurements were generally high, sometimes up to 50% higher than the actual value, and it also seems that the higher measurements had greater error.

## **Temperature & Conductivity**

Temperature affects other parameters including conductivity and pH. The conductivity of a solution is dependent on the ions present within it. Therefore, as struvite crystals are formed in the crystallizer, the conductivity should increase. Temperature and conductivity were measured using the Oakton Instruments/Eutech Instruments PC 300 probe.

## **Total Suspended Solids (TSS)**

It was not known how the TSS would influence the operation of the crystallizer. Solids provide extra nucleation points for struvite to form and can cause clogging in the tubing and the reactor; this requires occasional shut-down of the reactor for clean-up. The TSS test was done approximately five days per week.

## **Batch Tests**

This experiment was typically carried out once a week and was designed to mimic the conditions in the anaerobic, anoxic, and aerobic stages of the treatment process, in order to determine the effect of returning the crystallizer effluent to the process. Two experiments were run simultaneously, one without crystallizer effluent, as a control, and one with crystallizer effluent. Protocols and method for the phosphate uptake and release batch test results for evaluation of the returned VFA are reported by Gibb (1995). The Dayton & Knight (2008) report provides further explanation of the test methods.

## DISCUSSION

The crystallizer was consistently run with an influent flow rate of 1.7 L/minute and a recycle ratio of 10. Influent magnesium and caustic flow rates were adjusted based on the centrate used and its characteristics. In general, the reactor operation was acceptably smooth, although there were some operational issues and that were different from what was anticipated. Initially, it was thought that there would be significant problems with clogging in the tubing to the reactor. However, this did not occur since additional effort was undertaken to lower the risk of high feed solids.

One of the steps taken to reduce solids in the influent was to allow the centrate to settle overnight, before using it in the crystallizer. This was very helpful in terms of removing solids and also allowed the centrate to cool to between 20°C and 29°C, before it was processed in the crystallizer. The disadvantage of this step is that some struvite precipitates out during storage and is not recovered due to the decrease in temperature. A controlled decrease in temperature is potentially available to cause the formation in struvite, in lieu of the use of caustic to increase in pH. Doing so could result in cost savings for chemicals. However, because of the importance of removing solids during this study and the time limitations, there was not an opportunity to fully investigate the temperature effect.

Although the reactor consistently produced struvite pellets, the size and quality of the pellets varied. Initially, pellets of up to 3.6 mm in diameter with high strength were produced but over the length of the study the average size was approximately 2 mm in diameter. The quality and size of pellets can be improved by optimizing the reactor operation. Figure 4 shows struvite formed during each of the first five weeks of the study.

**Figure 4 - Pellets formed during the study**



Two important operational parameters are the Supersaturation Ratio (SSR) and the Magnesium: Phosphate ratio, which was maintained at 1.2:1. To confirm that the proper ratio is maintained, accurate measurements of magnesium and phosphate concentrations

must be available. The SSR must be high enough to ensure struvite nucleation, but not high enough that too many small particles are formed (fines).

A continuous mode of operation is desirable. Since the reactor recycles flow at ten times the influent flow rate, it could operate in “recycle mode” when new centrate was not available. When, on occasion, the reactor was completely shut down the crystals that were forming and fines that were in the reactor stuck together and were difficult to separate. This resulted in having to empty the contents of the reactor completely and start fresh. In order to “jump-start” the formation of struvite crystals, previously formed crystals were added to the reactor as seed. This greatly reduced the amount of time required to form new crystals.

## RESULTS

### Volatile Fatty Acids

On average, the volatile fatty acid (VFA) content of the influent to the crystallizer was approximately 1300 mg/L as acetic acid and the effluent from the crystallizer had a VFA content of approximately 900 mg/L as acetic acid (See Table 2 and Figure 4) . This shows that there is, on average, a drop in VFAs through the crystallizer of 24%. While the reduced concentration of VFAs in the effluent remains beneficial to the biological nutrient removal process of the plant, efforts to improve the recovery efficiency in the crystallizer may be warranted.

**Table 2 - Volatile Fatty Acid Content**

	Influent (mg/L as acetic acid)	Effluent (mg/L as acetic acid)	% drop through crystallizer
Overall avg.	1259	946	24%
Overall St. dev.	620	518	20%

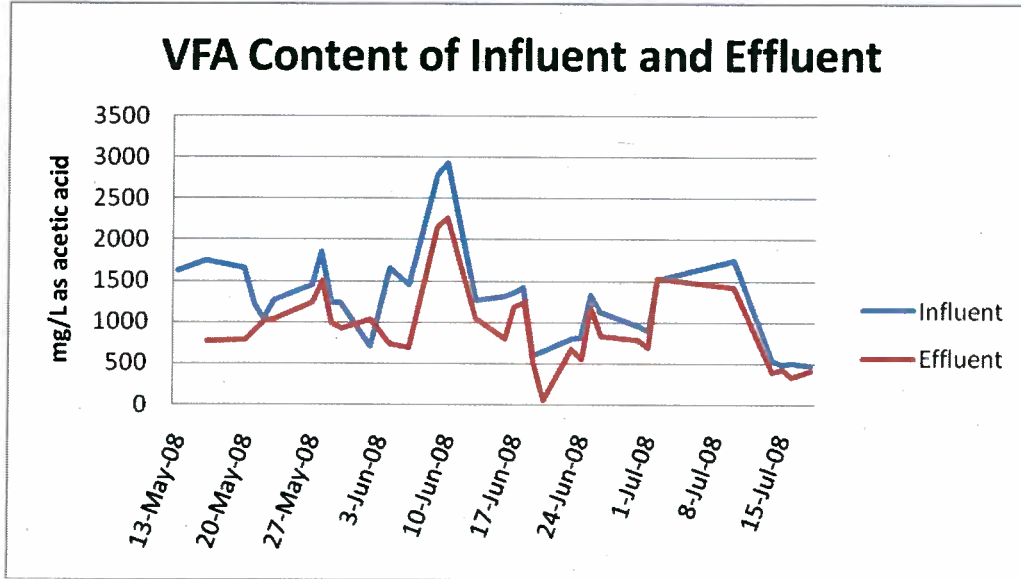
VFA was approximately 60% to 70% acetate and agrees with results given by Chu (1994) for the Salmon Arm ATAD. Propionate, butyrate and valerate make up the balance in essentially equal portions.

Storage also has an impact on the VFA content as there was an increase in VFA concentration during the storage of some batches, indicating that fermentation was occurring; while other batches showed a decrease in VFA concentration, indicating consumption was occurring. On average, there was an increase in the VFA concentration throughout the storage period of 7% from the beginning to the end of the batch.

The VFA production from the crystallizer averaged about 22.5 mg VFA/g VS. The loss of VFA from the struvite formation was higher than reported by Jiang (2007). Loss of the VFA could not be explained except that aeration in the crystallizer tower may have taken

place. The VFA production would be several times larger had the feed source been taken from the first cell of the ATAD reactors.

**Figure 5 - VFA Concentration of Crystallizer Influent and Effluent**



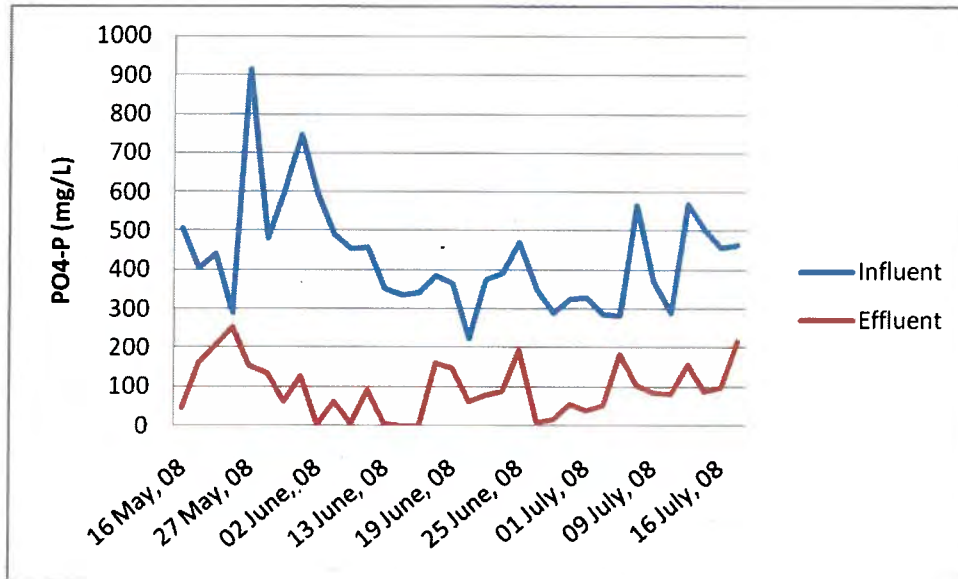
### Phosphate

On average the centrate from the ATADs, which was used as influent to the crystallizer had an orthophosphate concentration, as phosphorus of 433 mg/L and in the effluent the concentration was 92 mg/L. This gives an average phosphorus removal of 77% (see Table 3 and Figure 5). This amount of phosphorus removal is approximately the same as what has been found in other pilot studies with similar influent phosphorus concentrations (Prasad, 2007). Higher removals are however, believed possible with further optimization of the process.

**Table 3 - Phosphate Concentration and Removal**

PO <sub>4</sub> -P (mg/L)	Influent	Effluent	% P removal
Overall average	433	92	77%
Overall Standard deviation	142	68	19%

**Figure 6 - Crystallizer Influent and Effluent PO<sub>4</sub>-P Concentration**



The influent flow rate to the plant is 4600 m<sup>3</sup>/d with an influent phosphorus concentration of 7.5 mg/L. This gives a daily phosphorus load of approximately 34.5 kg/d. The pilot crystallizer would remove 2.4% of this load if operated continuously and a full scale reactor would remove approximately 26% of the total phosphorus load in the form of crystals. This phosphorus is removed in the form of a saleable fertilizer; its removal in this way would decrease the amount of sludge produced since alum addition would not be used to remove phosphorus prior to the centrifuge.

### Ammonium Nitrogen

Ammonium nitrogen was measured in the influent and effluent of the crystallizer

On average 34% of ammonium nitrogen (NH<sub>4</sub><sup>+</sup>-N) removal was achieved, based on an average influent NH<sub>4</sub><sup>+</sup>-N concentration of 871 mg/L. This removal is higher than is normally achieved in struvite reactors and is partially due to the high influent concentration. Normally, the molar ratio of N: P removal is approximately 1.1:1 however, in this study, a molar ratio of 1.7:1 N: P removal from the liquid was achieved. Several samples of struvite crystals were tested for their make-up, by weight of the three key elements (magnesium, nitrogen, and phosphorus). All samples had approximately a 1:1:1 molar ratio of P:N:Mg. This shows that there is an unaccounted for loss of nitrogen. It is unclear why this extra removal is occurring; one possible source is anaerobic oxidation of ammonia by Anammox bacteria. It is also possible that some of the extra ammonia removal is attributable to ammonia “stripping”, although any significant stripping would not normally be expected at pH operating ranges used in this study.

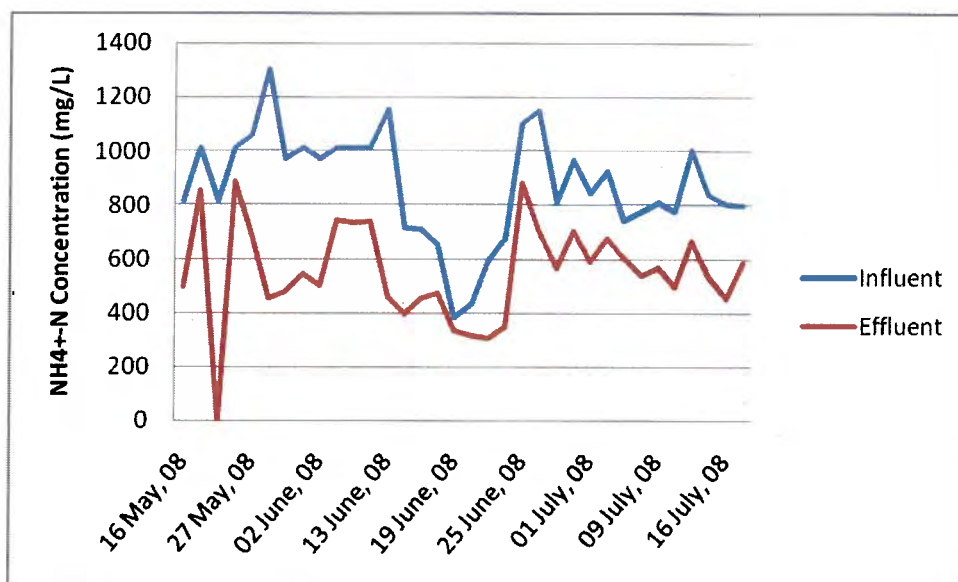
Table 4 shows the average influent and effluent ammonium-nitrogen concentration and the percent removal and their standard deviations. Figure 6 shows the change in influent

and effluent ammonium-nitrogen concentration throughout the study and it is clear that there is significant removal of nitrogen throughout.

**Table 4 - Ammonium Nitrogen Concentration and Removal**

NH <sub>4</sub> -N (mg/L)	INFLUENT	EFFLUENT	% REMOVAL
Overall avg.	871	566	34%
Overall st. dev.	199	157	13%

**Figure 7 - NH<sub>4</sub><sup>+</sup>-N Influent and Effluent Concentration**



### Batch Test Results

Batch tests were typically done weekly, to determine the potential effect of returning the VFA-rich crystallizer effluent to the primary tanks for improved nutrient removal. Several different configurations were used in order to better understand the effect it would have. Since the centrifuge was installed in 1998 the centrate from the ATAD treatment has been returned to the primary tank at the head of the plant. This has provided, in combination with the VFA in the influent sewage, sufficient fatty acids to achieve biological phosphorus (bio-P) removal.

The first set of batch tests done used a large volume of crystallizer effluent. This was done to show an exaggerated effect. Table 5 shows that the release during the anaerobic phase was sometimes greater in the control and sometimes greater in the experiment. However, the average phosphorus release is higher in the experiment than in the control and has a much lower standard deviation, indicating it is more consistent.

**Table 5 - Release during Anaerobic Phase**

PO <sub>4</sub> -P (mg/L)	12 May, 08	21 May, 08	27 May, 08	12 June, 08	Average	Standard Deviation
Control	1.4	0	22.1	16.8	9.9	11.1
Experiment	11.6	14.0	16.2	13.0	13.7	1.9

In order to ensure that there was no other source of VFAs that would interfere with the test results, subsequent batch tests were done using the full-scale ratios of inputs with the additional step that the sample from the primary tanks was pre-aerated. Before the samples were mixed and the test carried out, the sample from the primary tanks was aerated for an hour, to remove any VFAs that might have been in the sample. The primary tank normally has a low concentration of VFAs as there is some fermentation in the sewer line to the plant. However, by aerating the sample from the primary tank, we could ensure that the only acetate that was available to the PAOs was from the addition of the crystallizer effluent. This allowed us to accurately assess its effect.

Table 6 shows that, by adding an amount of crystallizer effluent that is proportional to what would be added at full scale, and removing all other sources of VFA, the VFAs from the crystallizer effluent improve the performance of the phosphorus removal process and provide more consistent operation. Overall, the number of tests completed is low because the study was short; it lasted only two months. However, the evidence suggests that substantial benefits for improvement can be made for implementing the use of the crystallizer and recovery of VFA at full-scale. These results also show that there are some VFAs in the plant influent as all the results are lower than those with no pre-aeration.

**Table 6 - Phosphorus Release with primary effluent pre-aeration**

PO <sub>4</sub> -P (mg/L)	11 Jul, 08	15 Jul, 08	17 Jul, 08	Average
Control	0	12	0	4
Experiment	9	8	7	8

## CONCLUSION

This was a demonstration project that supports a full scale application. The study provides evidence that at full scale the combination of fermenter/ATAD and struvite crystallizer could improve the performance of the nutrient removal process and the solids digestion process, eliminate the need for ferric or alum for phosphorus removal from the centrate, and provide consistent nutrient recovery in the form of a saleable end product.

At full scale, the diversion location of the digester liquor would be the first reactor, and concentrated solids from the separation of the liquid fraction could be returned to the ATAD system to improve digester performance. In the pilot study, to facilitate the existing process configuration, the diversion took place at the final reactor where VFA was a lower concentration. Nevertheless, the VFA recovery was significant. The use of a 2 day SRT ATAD reactor would improve the VFA recover significantly from 20 mg VFA/g VS to potentially 100 mg VFA/g VS. The majority of the VFA is acetate, and ranged from 60 to 70%.

Formation and recovery of struvite produces a saleable chemical fertilizer, and also avoids costly operational challenges of struvite removal from the dewatering centrifuge, related equipment and piping. It also benefits the treatment process by reducing the phosphorus and ammonium content of side streams that are returned to the wastewater treatment plant.

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