Utilising laboratory experiments as a first step to introduce primary sludge hydrolysis in full-scale

K. Jönsson, A. Pottier, I. Dimitrova and U. Nyberg

ABSTRACT

Laboratory experiments have been utilised as a tool to determine the possible yield of soluble COD and VFAs from settled influent and preprecipitated sludge at Klagshamn wastewater treatment plant and to determine the degradability of the organic matter. The release of ammonium and orthophosphate from the hydrolysed sludge has been estimated. It is possible to produce soluble organic matter of good quality from settled influent and preprecipitated sludge at Klagshamn WWTP. Denitrification rates of 3.1 mg NO_3—N/(g VSS × h) were found for the hydrolysate in laboratory tests. Owing to the low phosphate concentrations in the hydrolysate from preprecipitated sludge, it shows a potential for postdenitrification despite stringent phosphorus outlet demands. Calculations based on data gained from the laboratory experiments show that about 50% of the external carbon source used today can be saved with minor changes in the plant operation except that the hydrolysis has to be established. Based on these findings, full-scale experiments with internal hydrolysis in the primary clarifiers were scheduled in one of the two lines at Klagshamn WWTP for the summer of 2007.

Key words | denitrification, hydrolysis, laboratory tests, preprecipitation, primary sludge, VFA

K. Jönsson
A. Pottier
Water and Environmental Engineering at the
    Department of Chemical Engineering,
Lund Institute of Technology,
P.O. Box 124, SE-221 00 Lund,
Sweden
E-mail: karin.jonsson@vateknik.lth.se;
    tony_pottier@yahoo.fr

I. Dimitrova
U. Nyberg
Malmö Water and Wastewater Works,
SE-205 80 Malmö,
Sweden
E-mail: ivelina.dimitrova@malmo.se;
    ulf.nyberg@malmo.se

INTRODUCTION

Denitrification is performed by a diverse group of heterotrophic bacteria capable of reducing nitrate to nitrogen gas. The conversion requires access to biodegradable organic matter and the quality, or rather the degradability, determines the denitrification rate and hence in its turn the volume needed for denitrification. Theoretically the needed ratio between COD and nitrogen is 2.86, but in practice a consumption of 5–6 g COD/g N denitrified is often needed due to bacterial growth and losses e.g. because of oxygen entering the denitrification zone (Kristensen et al. 1992).

Influent wastewater often contains large amounts of organic matter, and COD/N ratios of 10 g COD/g N or higher are commonly found. However, a major part of the organic matter in wastewater is trapped in particles and hence not directly available for denitrification; the ratio between soluble COD and nitrogen is frequently only ca 4 g COD/g N (Henze et al. 1995). Primary settling and preprecipitation decrease the COD/N ratio as the major part of the nitrogen remains in the water phase while organic matter is removed by these processes. An efficient primary treatment is however favourable for the nitrification process due to less competition and also advantageous from an energy consumption perspective as less aeration is needed.

If the carbon available for denitrification is insufficient to meet the effluent demands, organic matter in form of external carbon source must be added. The dosing will enhance the process stability and flexibility as the addition can be adjusted to the actual needs of the process at every time. The drawbacks are the cost of carbon source and the extra sludge production.

An alternative is to apply sludge hydrolysis to produce a carbon source of good quality from the particles present in the wastewater, and subsequent dosing where and when

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needed in the process. According to a recent nationwide survey in Sweden 25 wastewater treatment plants add external carbon in order to facilitate their nitrogen removal processes, while seven use internal carbon sources from hydrolysis of sludge (Nicolić & Sundin 2006). Sludge hydrolysis has lately begun to gain enhanced interest amongst other things because of a perpetually increased price of external carbon.

There are basically two different ways to arrange primary sludge hydrolysis, see Figure 1. In the in-line alternative, primary sludge is kept in the primary clarifiers for a longer time than usual so that a sludge layer is created at the bottom. Anaerobic conditions will prevail in the sludge layer and hydrolysis will take place. In order to wash the formed soluble COD out of the sludge, the sludge is lifted to just below the water surface by a pump. The soluble COD will follow the water to the next treatment step and the sludge will settle again. The separation of the recirculated suspended solids will however not be ideal, leading to an increased load of suspended solid on the biological processes.

In the side-stream alternative, primary sludge is pumped to a separate tank for hydrolysis after which the formed soluble COD is separated from the sludge e.g. by centrifugation. Side-stream hydrolysis is easier to control compared to in-line hydrolysis as it is operated independently of the wastewater flow. The operator can choose where and when to dose the soluble carbon based on the needs of the biological processes and divert only the carbon needed for denitrification to the biological processes and the rest to the sludge treatment where it can be utilised for gas production if anaerobic digestion is applied, instead of giving rise to additional oxidation demands and increased sludge production in the biological processes.
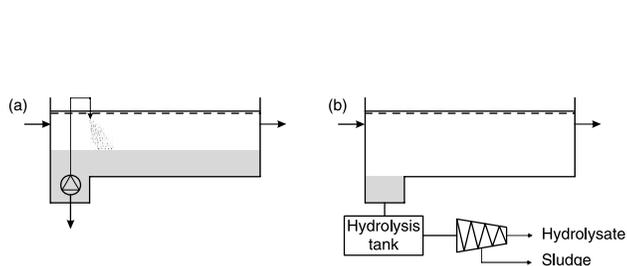
Aim of this study

The aim of this study was to use laboratory experiments as a tool to determine the possible yield of soluble COD and VFAs from preprecipitated sludge at Klagshamn wastewater treatment plant and to determine the degradability of the organic matter. The release of ammonium and orthophosphate from the hydrolysed sludge would be estimated. Based on data gained from the laboratory experiments, estimations of the effect of implementation of a full-scale primary sludge hydrolysis process at Klagshamn wastewater treatment plant would be performed.

Klagshamn wastewater treatment plant

The Klagshamn wastewater treatment plant was upgraded for extended nutrient removal in 1996–1997. The selected upgrading concept was based on full-scale and pilot-plant studies (Andersson *et al.* 1998; Aspegren *et al.* 1998). By efficient preprecipitation the organic load on the activated sludge plant is low and nitrification is maintained in small volumes. Denitrification is mainly accomplished in a moving bed biofilm reactor (MBBR) with addition of external carbon source. To reduce the load on the MBBR process, the first zone in the first part of the activated sludge process is operated anoxic to make use of the small amount of carbon of good quality that remains in the water after the preprecipitation together with the nitrate recirculated via the return sludge. The current process layout is outlined in Figure 2. Tables 1 and 2 give the basic technical information of the plant, the characteristics of the influent and the discharge requirements.
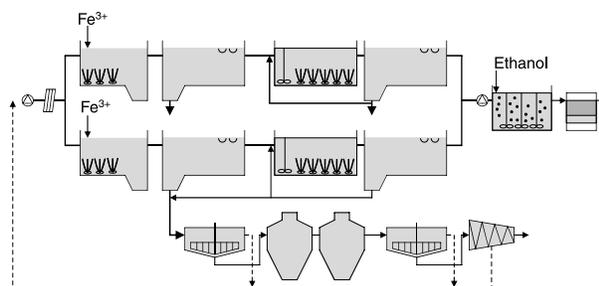

Figure 1 │ Primary sludge hydrolysis: (a) in-line configuration and (b) side-stream configuration.

Figure 2 │ Process layout of Klagshamn wastewater treatment plant.

Table 1 | Technical information of Klagshamn wastewater treatment plant

	Number	Area (m²)	Volume (m³)
Primary clarifiers	4	4 × 250	4 × 550
Activated sludge basins	2*	–	2 × 2200
Secondary clarifiers	8	8 × 170	8 × 610
MBBR	2	–	2 × 550
Dual media filters	5	220	–

*Each basin is divided in 8 cells, each 550 m³.

Table 2 | Characteristics of the influent, internal sampling and discharge limits at Klagshamn WWTP

	Influent	After preprecipitation	Before MBBR	Discharge limits
Flow (m³/d)	18,000	–	–	–
COD (mg/l)	200–500	–	–	–
BOD$_7$ (mg/l)	100–150	30–50	5–10	10*
Tot-N (mg/l)	20–40	20–35	15–20	12†
Tot-P (mg/l)	3–7	1–2	0.5–1.0	0.3*
Temperature (°C)	–	5–16	–	–

*Monthly average.
†Yearly average.

MATERIALS AND METHODS

Hydrolysis experiments

Preprecipitated primary sludge collected at Klagshamn WWTP was kept in 1.8 litres, gently stirred reactors at 20°C and 10°C, respectively, for 10 days. Nitrogen gas was introduced in order to ensure strictly anaerobic conditions. As a comparison, settled sludge from incoming water collected just before the bar screens was hydrolysed in the same type of reactor at 20°C. The experimental set-up is summarised in Table 3. Samples were withdrawn from the reactors every day and analysed for pH, VFA, COD, NH_4-N and PO_4-P. The degradability of the organic

matter was also examined using denitrification tests. These so-called nitrate uptake rate (NUR) tests were performed on the fourth day of the experiments.

Analysis methods

All measurements of COD, VFA, ammonium and ortho-phosphate have been carried out for filtered samples. Cellulose filters No. 1002 with a filtering rate of 250 ml/min were used for all filtration of samples. COD, ammonium, nitrate, nitrite and orthophosphate were analysed using Dr Lange tests LCK 114, LCK 303, LCK 339, LCK 341 and LCK 348/349, respectively. All readings of Dr Lange analyses were performed using a Dr Lange LP2W. SS and VSS were analysed according to *Standard Methods*. VFA was analysed by means of chromatography, Agilent 6850 Series GC System. A WTW 320 pH-meter was used for all measurements of pH. NUR tests have been performed according to Kristensen *et al.* (1998).

RESULTS AND DISCUSSION

Based on the daily samples withdrawn from the hydrolysis reactors, the production of VFAs and soluble COD related to the VSS concentrations in the reactors were calculated, see Figure 3. Data from similar experiments with prepre-cipitated sludge from another treatment plant is also included in the figure as a comparison. It is seen that the yields of soluble COD and VFAs for the hydrolysis of preprecipitated sludge at 20°C is similar for the two sludges originating from different treatment plants. The yield at 10°C is however clearly lower in these experiments compared to the corresponding experiments made by Daton & Wallergård (2003). Both sludge types in these experiments contained VFAs and soluble COD to some

Table 3 | 3 Experimental set-up of the hydrolysis reactors

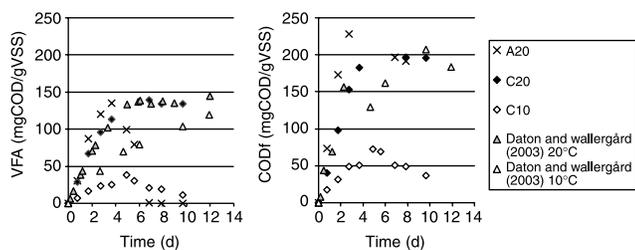| Experiment	A20	C20	C10
Sludge type	Sludge collected before bar screens	Preprecipitated sludge	Preprecipitated sludge
Initial volume in reactor (l)	1.8	1.8	1.8
Temperature in reactor (°C)	20 ± 0.1	20 ± 0.1	10 ± 0.1
Initial VSS in reactor (g/l)	5.0	16.8	16.8

Figure 3 | Production of VFAs and soluble COD related to the VSS concentrations in the reactors. As a comparison results obtained with prepreciptiated sludge from another WWTP are also included.

extent already before the start of the hydrolysis reactors, ranging from 7 to 14 mg COD_{VFA}/g VSS and 33–40 mg COD_f/g VSS.

Maximum yield of soluble COD related to VSS or COD in the reactor is given in Table 4. A factor of 1.42 g COD/g VSS has been used for the translation from VSS to COD units (Henze *et al.* 1995).

For precipitated sludge it was possible to solubilise 7% of the total COD at 10°C and 16% at 20°C. For the sludge without chemicals the corresponding figure was slightly higher, 19%. Jørgensen (1990) and Brinch *et al.* (1994) found that 10–15% of the total COD consisted of soluble COD at 10–30°C and 15–25°C, respectively.

The results from the A20 experiment indicate that sludge without precipitation chemicals might be faster to hydrolyse and that the yield per VSS might be bigger. The initial hydrolysis rate is almost twice as high for the sludge without chemicals. This contradicts the results of Jørgensen (1990) as he only found a minor effect caused by chemicals.

In contrast to the higher yield of soluble COD for the sludge without chemicals, the ratio between VFAs and

Table 4 | Maximum yield, VFA/COD ratio, initial hydrolysis rate and COD to nutrient ratios

Experiment	A20	C20	C10
Maximum yield (mg soluble COD/g VSS)	(269)*	229	105
Maximum yield (mg soluble COD/total COD)	19%	16%	7%
Average VFA/soluble COD ratio in hydrolysate	43%	60%	46%
Initial hydrolysis rate (mg soluble COD/(g VSS × h))	4.2	2.3	0.73

*Missing data for the A20 reactor, thus the maximum yield might be higher.

soluble COD in the hydrolysates is higher for the pre-precipitated sludge at 20°C (60%) compared to the sludge without chemicals (43%), see Table 4. The results for the preprecipitated sludge are in good agreement with previous results. Kristensen *et al.* (1992) found that VFA-COD made up 60% of the total COD in centrifuged hydrolysate. Æsøy & Ødegaard (1994) found that on average 66% of the soluble organic matter was VFAs for hydrolysis at 37°C of a sludge comprising of 70% chemical sludge and 30% biological sludge. Jørgensen (1990) found 60–80% of soluble COD as VFA when preprecipitated sludge was hydrolysed. In this study the VFA/CODf-ratio was lower (46%) at lower temperature. The same trend of tempera-ture-dependent composition of the produced soluble COD has also been found in a previous study on return sludge hydrolysis (Jönsson & Jansen 2006). Daton & Wallergård (2003) found ratios of 40–45% for hydrolysis of prepreci-pitated sludge at both 10°C and 20°C. The distribution between VFAs and soluble COD other than VFAs in the hydrolysate is usually not crucial if the hydrolysate will be utilised as easily degradable carbon for facilitating deni-trification. However, if the purpose is to use the carbon source for enhanced biological phosphorus removal, the quality of the soluble COD, or in other words the available amount of VFAs, is important.

After four days of hydrolysis, hydrolysate was with-drawn from the reactor operated at 20°C with preprecipi-tated sludge in order to determine the denitrification rate possible to achieve (NUR test). Acetate was used as a reference carbon source and the test was performed at 20°C. The addition of hydrolysate as well as acetate both resulted in denitrification rates of 3.1 mg NO_3—N/(g VSS × h). The equal denitrification rates were expected as the hydrolysate contains a large amount of VFAs. Brinch *et al.* (1994) also found equally high denitrification rates for acetate and hydrolysate in their experiments. It is well known that denitrifying bacteria need to adapt to some specific carbon sources, e.g. methanol, to be able to utilise it. Studies made by Aravinthan *et al.* (2001) indicate that there also might be a factor of acclimatization for a denitrifying bacterial culture to a carbon source in the form of hydrolysate. They showed that after a week of feeding with the hydrolysate in question, the acclimatized sludge gave rise

to higher denitrification rates compared to non-acclimatized sludge.

The main reason for performing sludge hydrolysis is to enhance the biological removal of nitrogen and phosphorus. However, during hydrolysis both ammonium and phosphate are solubilised from the sludge. Therefore, the hydrolysis should not lead to too big a release of nutrients because the removal of nutrients will increase oxygen consumption, consumption of the easily biodegradable carbon of the hydrolysate and in worst case increase the concentrations in the final effluent. For this, the COD/P and COD/N ratios in the hydrolysate are important factors to consider. The impact of solubilisation of nutrients is often disregarded when evaluating hydrolysis processes. In literature can be found that Æsøy and Ødegaard (1994) experienced that 0.016 g NH_4—N/g VS was released and in a series of experiments performed by Bouzas *et al.* (2007) the increase of phosphate in pretreated wastewater compared to influent water due to the hydrolysis never exceeded 1 mgP/l and the corresponding ammonia increase ranged from 0.3 to 3.8 mg N/l. Jørgensen (1990) found that 1 mg NH_4—N was released from the sludge per 10–30 mg of solubilised COD and that only trace amounts of orthophosphorus could be measured, as the hydrolysed sludge in this case also contained precipitation chemicals that took care of the phosphorus. In the present investigation the resulting PO_4—P concentration in the hydrolysate from preprecipitated sludge was negligible, while the solubilisation from the sludge without chemicals was up to 0.025 mgPO_4—P/mg COD solubilised. Regarding nitrogen, a common linear relationship between solubilised COD and ammonium was found for all three reactors; 0.035 mg NH_4—N/mg COD solubilised, i.e. the hydrolysate has a good COD_f/NH_4—N ratio of 29.

Potentials for implementation of primary sludge hydrolysis at Klagshamn wastewater treatment plant

Hydrolysate from primary sludge hydrolysis can be added to facilitate both pre- and postdenitrification at Klagshamn WWTP. The production of preprecipitated sludge at Klagshamn WWTP is ca 6,000 kg TS/d. Using data from the hydrolysis experiment at 20°C and a retention time of 2.5 days, 150 mg soluble COD/g VSS is possible to produce.

The amount of soluble COD present in the sludge before the start of the hydrolysis experiment (ca 30 mg soluble COD/g VSS) should also be taken into account. The VSS/SS ratio in the preprecipitated sludge is 78% and hence the production of soluble COD is 840 kg/d. Considering losses due to that only ca 80% of the hydrolysate can be separated from the sludge, 675 kg soluble COD/day is a reasonable estimation of the possible production. Today, external carbon sources corresponding to 1230 kg COD/day is on average added to the postprecipitation plant at Klagshamn WWTP. Hence 55% of the external carbon need can be substituted by hydrolysate. Bouzas *et al.* (2002) gives examples of WWTPs where between 30 and 90% of the need of external carbon could be covered by sludge hydrolysis.

There is room enough in the activated-sludge basins to use between 12.5 to 25% of the volume as anoxic zones. With the present process configuration and operation the predenitrification will however be carbon limited, why addition of hydrolysate to the preprecipitation process might be wise. The total anoxic volume available would be 2200 m^3, the MLSS concentration 2.0 kg SS/m^3 and the VSS/SS ratio ca 80%. The nitrified nitrogen in the return sludge is ca 0.015 kg N/m^3 × 18000 m^3/d = 270 kg N/d as the applied return sludge ratio is ca 100%. If the 675 kg soluble COD is added to the predenitrification zone, ca 125 kg N/d can be denitrified. The minimum denitrification rate needed, assuming 25% anoxic volume, is hence 1.5 mg N/(g VSS × h), which is half as much as the denitrification rate obtained with the hydrolysate at 20°C.

Adding hydrolysate to pre- or postdenitrification: advantages and drawbacks

At Klagshamn WWTP it is very easy to establish in-line hydrolysis in the existing primary clarifiers. The hydrolysate is dosed direct to the existing anoxic compartment where sufficient nitrate is available due to the internal sludge recirculation. The only drawback in this concept is that the increased addition of organic material will lead to higher sludge production. This leads to a reduction of the very high nitrification rate of today where preprecipitated wastewater with a BOD_7/Tot-N ratio just above 1 is very favourable for fast nitrification. The addition of hydrolysate has to be limited to a level where the existing tank volumes enable full

nitrification and at the same time complete utilisation of the added carbon.

Separate hydrolysis has the advantage that an almost particle-free hydrolysate can be produced and dosed when necessary. The dose can be made to the activated sludge system but also to the MBBR as a replacement of some of the ethanol used today. Any particles left in the hydrolysate will just pass the denitrification stage and be separated in the dual-media filters. The low phosphate and ammonium content in the hydrolysate is not expected to cause any problems for the fulfilment of the discharge limits.

The calculation shows that about 50% of the external carbon source can be saved with minor changes in the plant operation except that the hydrolysis has to be established. Based on these findings, full-scale experiments with internal hydrolysis in the primary clarifiers are scheduled in one out of the two lines at Klagshamn WWTP for the summer of 2007. The aims are:

- to examine whether in-line primary sludge hydrolysis can be utilised during at least the summer period to partly replace the external carbon.
- to estimate the effect in terms of process rates and costs of the extra load of carbon, ammonium and suspended matter to the activated sludge tank.
- to characterise the quality of the hydolysate with respect to particles, phosphorus and ammonium in order to evaluate the possibility to add hydrolysate direct to the postprecipitation plant (MBBR).
- to evaluate the potential loss of gas from the digesters since cost reduction due to utilisation of hydrolysis for improved nitrogen removal has to be balanced against loss of carbon fed to the digesters.

CONCLUSIONS

It is possible to produce soluble organic matter of good quality from settled influent and preprecipitated sludge at Klagshamn WWTP. Denitrification rates of 3.1 mg $NO_3-N/(g\ VSS \times h)$ was found for the hydrolysate in laboratory tests.

The production of hydrolysate is estimated to be sufficient for replacement of about 50% of the ethanol

used today in a predenitrifying stage with minor change in plant operation.

Due to the low phosphate concentrations in the hydrolysate from prepecipitated sludge, it makes up a potential for postdenitrification despite stringent phosphorus outlet demands.

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