

## Concentration of nutrients from urine and reject water from anaerobically digested sludge

M. Ek\*, R. Bergström\*, J.-E. Bjurhem\*, B. Björlenius\*\* and D. Hellström\*\*

\*IVL Swedish Environmental Research Institute, Box 21060, SE-100 31 Stockholm, Sweden  
(E-mail: mats.ek@ivl.se; jan-erik.bjurhem@ivl.se; rune.bergstrom@ivl.se)

\*\*Stockholm Water Co., SE-106 36 Stockholm, Sweden (E-mail: berndt.bjorlenius@stockholmvatten.se; daniel.hellstrom@stockholmvatten.se)

**Abstract** Experiments with concentration of nutrients from source separated urine and reject water from digestion of sludge in sewage treatment plants (STP) have been performed in laboratory and pilot scale. The methods tested were membrane filtration with reverse osmosis (RO), evaporation, and precipitation of phosphorus and distillation of ammonia. In membrane filtration, pre-filtration with particle separation at 5–10 µm was enough to avoid clogging of the membranes. Separation of phosphorus (P), potassium (K) and sulphur (S) was almost 100%, while separation of nitrogen (N) was dependent on pH. The capacity of flux increased with temperature and pressure. In evaporation, all P, K and S were still in the concentrate, while pH had to be decreased to 4.5 to avoid significant loss of N. In precipitation and distillation, about 90% of P could be recovered from urine as magnesium ammonium phosphate (MAP) just by adding MgO. For the reject water pH was first increased by aeration to remove CO<sub>2</sub>. Ammonium can be distilled from the water phase after precipitation of MAP, without further increase of pH. At least 80–90% of N can be distilled in 5–10% of the total volume. The article also discusses the quality of different products, cost of separation, and energy and chemical demand.

**Keywords** Nutrient recovery; separation; sewage reject water; urine

### Background

There are at least two good reasons to try to separate streams with high concentrations of nitrogen and phosphorus from the influent to sewage treatment plants (STP). First, the load on the STP, and thus the cost, will be lower. Secondly it will increase the possibility to recirculate the nutrients to farmland. Two streams with high concentrations of nutrients are source-separated urine and reject water from anaerobic digestion of sludge. Separated urine contains a great part of the total nutrients in normal sewage; 80% of N, 55% of P, and 60% of K in just 1.5% of the volume of the sewage (Jönsson *et al.*, 2000). In reject water, the concentration of N is about 30 times higher than in sewage, and the same is valid for P after digestion of sludge from biological phosphorus reduction (BioP). Many attempts have been made to concentrate or extract nutrients from these streams. Membrane filtration has been discussed (Ericsson and Isaksson, 1993; Jönsson *et al.*, 2000), as well as evaporation or drying (Thurdin, 1997). Specific precipitation of P has been investigated (Woods *et al.*, 1999; Jönsson *et al.*, 2000; Lind *et al.*, 2000; Battistoni *et al.*, 2001; Jaffer *et al.*, 2001; Ueno and Fujii, 2001). Stripping or distillation of ammonia (Thøgersen, 1992; Siegrist, 1996; Janus and van der Roest, 1997; Katehis *et al.*, 1998; Jönsson *et al.*, 2000) and adsorption of ammonium in ion exchangers (Lind *et al.*, 2000) have been reported. In this work, membrane filtration, evaporation, and precipitation/distillation of urine and reject from digestion of

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BioP sludge are compared. Yield of nutrients in different form, use of energy and chemicals, and economical possibilities are compared.

## Materials

### Urine and reject water

Urine was collected from Understenshöjden, a residential area with urine separating toilets. It was brought to the laboratory in 600 l polyethylene containers and stored at 4 °C until used in experiments. Table 1 shows some characteristics of the urine used, compared to “normal” urine from the same area (Jönsson *et al.*, 2000). Reject water from digestion of sludge from BioP was not available in Sweden. To simulate a BioP reject, reject water from a STP with conventional precipitation with iron was used after addition of extra P. Addition of phosphate gave a precipitate with residual metals, and also some particles settled. This precipitate was removed, and the half-synthetic reject water was stored at 4 °C until used. Table 1 shows the composition of the reject water used in the experiments.

It is obvious that the urine samples used were more diluted than normally, either because of excess flushing or leakage of drainage water into the system. In all calculations of cost and use of resources data have been recalculated to a “normal” source separated urine containing 3600 g N/m<sup>3</sup>, 310 g P/m<sup>3</sup>, 900 g K/m<sup>3</sup>, and 300 g S/m<sup>3</sup>.

### Separation methods

*Pre-treatment before RO.* To avoid clogging of the membranes with particles, three different methods were used; sieving through 0.5 mm sieve, filtering with 5 µm cartridge filter, and ultra-filtration (UP) with cut-off at 100,000 Dalton. The pre-treatments were evaluated by determination of the flux through the spiral wound membrane.

*RO filtration.* Tests were made in commercial membrane units in pilot scale. Both spiral wound and tube membranes were used, and influence of pre-treatment, pH (with different acids), pressure, and temperature were studied. In each test, the water was first circulated over the membrane during at least several hours before concentration started. This was done to get stable data on the flux as l/(m<sup>2</sup> \* h). After this concentration was made, normally to half the volume, Volume Reduction Factor (VRF) 2, water was again circulated to give a stable flux. Concentration continued in the same way, until maximum VRF regarding the decreasing flux was reached. Table 2 gives some data for the membranes used. Both were made from polyamide and separated more than 99% of NaCl.

**Table 1** Composition of the tested urine and reject water

Parameter	Unit	Tested urine	“Normal” urine	Reject water
pH		9.2–9.3	9.1	7.9
Conductivity	mS/cm	15–20	25	5.8
Total solids	mg/l	4400–7000	8100	2100
Ash	mg/l	3500–4800	5800	1100
Suspended solids	mg/l	51–73	–	100
COD	mg/l	2200–2800	2100*	270
TOC	mg/l	910–1200	–	110
Total P	mg/l	170–240	310	130
PO <sub>4</sub> -P	mg/l	160–230	–	130
Kjeldahl-N	mg/l	2200–3200	3600	820
NH <sub>4</sub> -N	mg/l	2000–2900	–	740

\*BOD<sub>7</sub>

**Table 2** Characteristics for the membranes used

Type of membrane	Max pressure Pa	Max temperature °C	pH range	Area m <sup>2</sup>
PCI AFC-99 tube	7	70	2–11	0.9
Filmtec SW30-HR spiral	6.9	45	2–11	2.1

*Evaporation and drying.* Samples were evaporated under vacuum in glass equipment at different pH. The condensate was collected for determination of mass balance for N. Concentrates from evaporation and RO were dried in a ventilated oven at increasing temperatures from about 50 °C.

*Precipitation of phosphate.* Phosphate was precipitated with Ca(OH)<sub>2</sub> or MgO in glass equipment in volumes between 0.8 and 5 litres. pH in the urine was already high enough for precipitation of MgNH<sub>4</sub>PO<sub>4</sub>·6H<sub>2</sub>O (MAP), but pH in the reject water had to be increased. This was performed by aeration (removal of dissolved carbon dioxide) or addition of NaOH. The precipitate was separated by sedimentation, but also sometimes with filtration. Tests were made with different amounts of added chemicals, pH, and mixing procedures.

*Distillation of ammonia.* Ammonia was distilled in glass equipment with reflux and collection of distillate in H<sub>2</sub>SO<sub>4</sub>. Tests were made at different pH, reflux, and amounts of distillate.

#### Analyses

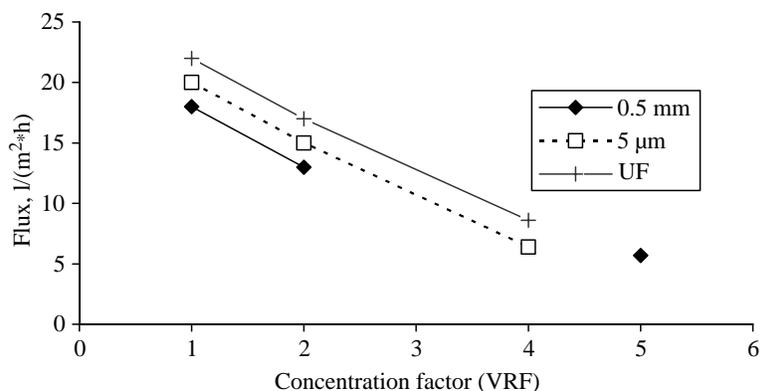
COD, NH<sub>4</sub>-N, and PO<sub>4</sub>-P were determined by ampoule-tests (Dr Lange), while TOC, total P, Kjeldahl-N, suspended solids, dry matter and ash were determined with standard methods. pH and conductivity were measured with electrodes, and metals were analysed with ICP.

## Results and discussion

### Concentration with reverse osmosis (RO)

Figure 1 shows the influence of different pre-treatments on flux.

Better removal of particles increased the flux, but the difference was not high enough to motivate the more expensive ultra filtration. Relevant pre-treatment should be determined by the amount of suspended solids, and aim to remove particles larger than about 5–10 µm. Cartridge filter with 5 µm was used in the further experiments. The two membranes gave similar results. AFC had a slightly better separation of N, and about 20% higher flux, but the much lower price for SW or a similar spiral wound membrane makes

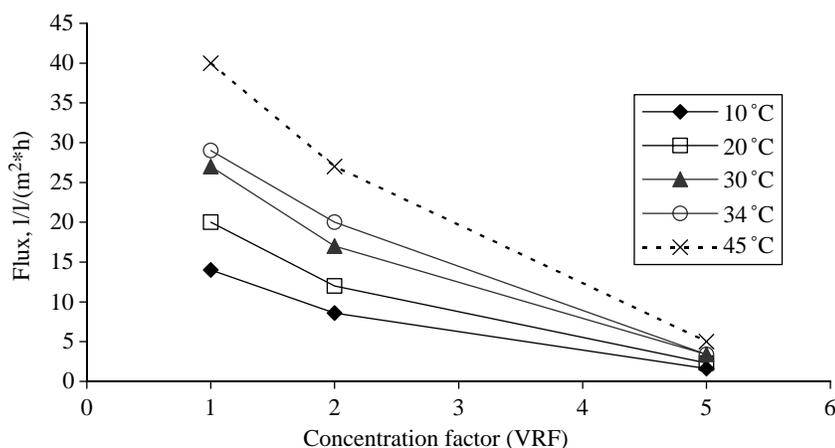
**Figure 1** Flux as a function of VRF and pre-treatment for urine

this more economic. 5 Pa trans-membrane pressure gave almost 50% higher flux than 4 Pa for urine at VRF 2. This motivates the somewhat more expensive equipment and higher pump energy use at 5 Pa, and this was used in most of the experiments. pH influenced both flux and separation. Table 3 shows some data from short tests with urine.

The influence on flux is probably dependent on the amount of extra salt added with the acids. The difference of performance increased with VRF. Sulphuric and nitric acids were tested for pH adjustment, and since the cheaper sulphuric acid did not cause any extra problems with the precipitation. This was used in most experiments. To decrease pH of normal urine to a value of 7, about 6 kg 96% H<sub>2</sub>SO<sub>4</sub> per m<sup>3</sup> are needed, and this increases the salt content from about 6 to 9 g/l. pH 6 demands about 11 kg acid and gives 13 g salt/l. In the final long running experiment (21 days) the separation differences at different pH could still be seen, but the separation was better. The concentrate after reaching VRF 5 with pH about 7 contained 95% of the total N and 99.7% of the total P. The mean flux to reach VRF 5 was about 15 l/(m<sup>2</sup> \* h) at 30 °C. Optimum of pH has to be determined in each case based upon the amount of N recovered and the cost for RO to reach a certain VRF. The risk of precipitation of MAP or CaCO<sub>3</sub> on the membranes at higher pH is also an important factor. The economical example here is calculated for pH 7. The greater loss of N to the permeate at higher pH, is probably due to a better separation of NH<sub>4</sub><sup>+</sup> than NH<sub>3</sub>. P is very effectively separated by the membranes, and the same is valid for K. The concentration of the reject water started with just some addition of H<sub>2</sub>SO<sub>4</sub>, but more had to be added during concentration. This is probably due to loss of carbon dioxide. The total addition was about 2.9 kg H<sub>2</sub>SO<sub>4</sub> per m<sup>3</sup> reject water when VRF 19 was reached. Separation of N was similar to that for urine, and the flux was higher. At the end of the long running test (18 days) a considerable amount of salt was found at the bottom of the concentrate tank. Chemical analyses showed that it was mainly MAP and some calcium phosphate. The average value of the flux to reach VRF

**Table 3** Capacity and separation at different pH (urine, 5 Pa, and 29 °C)

pH	Flux at VRF 5 l/(m <sup>2</sup> ·h)	N in concentrate % of total	P in concentrate % of total
9.2	10.5	79	> 99.9
7.0	7.2	91	> 99.9
6.0	5.5	98	> 99.9



**Figure 2** Flux as a function of temperature and VRF (urine, SW-membrane, pH 6.5, and 5 Pa)

19 at about pH 7 was  $35 \text{ l}/(\text{m}^2 \cdot \text{h})$  at  $35^\circ\text{C}$ . Temperature had a strong influence on the flux (Figure 2), but almost none on the separation. If it was possible to work at  $45^\circ\text{C}$  instead of  $20^\circ\text{C}$ , only half the membrane area would be needed and the final concentration could be a slightly higher. Urine is normally stored below ground at the site of collection, and the temperature is therefore, in Sweden, between  $5$  and  $15^\circ\text{C}$  most of the year. However, it was shown that even at an energy price of  $0.4 \text{ SEK}/\text{kWh}$  it is economically beneficial to heat the urine before RO treatment. Reject water collected directly after digestion and following separation will have a temperature of around  $35^\circ\text{C}$ . The RO treatment of  $1 \text{ m}^3$  urine will produce 200 litres of concentrate at pH 7 will contain 95% of the nitrogen, 90% of the phosphorus (some losses in the pre-filtration), almost all other salts, and also non desired compounds like heavy metals, hormones, and drug residues. There will be 800 litres of permeate, containing mainly ammonium. The concentration of N will be about  $200 \text{ mg}/\text{l}$ , and it should be treated together with the sewage.  $1 \text{ m}^3$  of reject water will produce 50 litres of concentrate at pH 7 containing 90% of the N and P and almost all other components in the reject water. The 950 litres permeate will be recirculated to the sewage treatment plant.

#### Evaporation and drying

Preliminary experiments showed that the value of pH should be kept below 6 to give more than 70% of N in the concentrate of the urine. In the final experiments, pH 4.5 and 5.5 were compared, and adjusted with  $\text{H}_2\text{SO}_4$ . The evaporation was driven to VRF 20, i.e. 5% concentrate. This could be done without any addition of antifoam agent. The temperature at the beginning was  $30^\circ\text{C}$ , but had to be increased during the evaporation. The result of this was that some material burned into the glass surfaces. pH 5.5 gave 89% and pH 4.5 gave 95% of N in the concentrate. The difference in yield of N resulted in about the same economical value as the extra  $\text{H}_2\text{SO}_4$  needed to reach pH 4.5 (12 and  $13 \text{ kg H}_2\text{SO}_4/\text{m}^3$  urine respectively). To further decrease the load of N to the sewage treatment plant, pH 4.5 was selected in the calculations.  $1 \text{ m}^3$  of urine would give 50 litres of concentrate with 95% of N and almost all other components. Further drying after evaporation or RO treatment gave a very hard cake of salt with a very unpleasant smell, and no additional experiments were made with this. The results from evaporation of reject water were very similar to the results from urine evaporation. pH 5.5 resulted in 86% N and pH 4.5 resulted in 95% of N in the concentrate at VRF 20. Concentration could be extended to reduce the volume even more.  $1 \text{ m}^3$  of reject water would produce 20 litres as a minimum value of concentrate with about 95% of N and almost all other components.

#### Precipitation of phosphate and distillation of ammonia

Both stored urine and reject water contain a lot of carbonate. Addition of  $\text{Ca}(\text{OH})_2$  to precipitate phosphate therefore results in a massive precipitation of  $\text{CaCO}_3$ .  $2.8 \text{ kg Ca}(\text{OH})_2/\text{m}^3$  was needed to precipitate 90% of P in urine, and this resulted in 150 litres of sludge. Addition of  $0.6 \text{ kg MgO}/\text{m}^3$  gave a much more compact precipitate of MAP ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ), in this case about 9 litres of sludge and still 90% precipitation of P. This corresponds to 1.5 times the theoretical amount of Mg. With filtration or sedimentation with long hydraulic retention time, the yield increases to 95% and the dry matter content is more than 15%. Addition of extra alkali had no effect, and different attempts to increase the crystal size (rods about  $4 \mu\text{m}$  in diameter and the length  $20 \mu\text{m}$ ) were not effective. pH after precipitation of urine with MgO was 9.2–9.3. Preliminary tests showed that pH had to be increased to at least 10 to obtain most of the ammonia in the distillate. This would give a high cost for NaOH (about  $10 \text{ SEK}/\text{m}^3$ ). However, tests with more functional columns showed that 80–90% of the ammonia could be distilled

resulting in only 5–10% of the total volume without increase of pH. This means that it is possible to get MAP as one product, containing almost 95% of P and 3% of N in less than 10 litres of crystal/urine suspension and about 85% of N in less than 100 litres of distillate. Both phases can be further upgraded and are virtually free from contaminants. The reject water has a lower pH, so direct precipitation with MgO is not possible. However, only aeration without addition of MgO increased pH to 8.7–8.8. There was a precipitate in the reject water of half-synthetic BioP with phosphorus salts and a lot of the organic suspended material. This would not be the same in a real BioP reject. After separation of this precipitate, MgO was added. In this case just 80% of the theoretical demand of Mg was added, to avoid an excess of Mg entering the sewage treatment plant giving problems with MAP in the equipment. About 65% of the residual P after aeration was precipitated as fine crystals similar as in the urine. The two precipitates were analysed, and the results are given in Table 4.

Data shows that the second precipitate is probably mainly MAP with a low content of heavy metals (showing cadmium as an example). As mentioned before, this was not a real BioP reject, but the results indicate that suspended solids should be removed before precipitation of MAP to give a relatively pure product. Distillation directly after the separation of the precipitates, without further increase of pH, gave more than 80% of the ammonia in 10% of the volume. The distillations were carried out at atmospheric pressure. Since it was not a genuine BioP reject, the data has to be taken with some caution, but the results indicated 3 litres of a MAP-like product/m<sup>3</sup> reject, containing 65% of P and 5% of N. The process also produces about 100 litres almost pure ammonia. The rest is recirculated to the STP.

#### Yields, resources used, and costs

Table 5 summarises the amounts of nutrients recovered.

For storage and transportation a low volume is important. The concentrates from evaporation have a low pH and probably have to be added to farmland together with lime. Important impurities in urine can be hormones and drug residues, while in reject water heavy metals are probably more important. Table 6 shows the main resources used, and cost estimates. Cost estimates are based on data from manufactures 2002 for different equipment and 20% annuity, 1000 SEK/ton 96% H<sub>2</sub>SO<sub>4</sub>, 2800 SEK/ton MgO, 20 SEK/kg membrane and evaporation cleaning agent, 0.4 SEK/kWh electric energy, 0.2 SEK/kWh heat energy, 200 SEK/h for manpower, and a maintenance of 2% of the investment cost. Costs were estimated for two different capacities, 2 and 10 m<sup>3</sup>/h, and continuous treatment. From Table 6, it is obvious that scale has a great influence on price in this range. Only RO treatment gives a relatively large volume of concentrate, due to the amount of salt. It is possible to reach the concentrate volumes of the evaporation in a more cost effective manner by combining RO at pH 7 and evaporation at pH 4.5. The estimated cost at 10 m<sup>3</sup>/h is 44 SEK/m<sup>3</sup> for urine and 19 SEK/m<sup>3</sup> for reject water. However, the

**Table 4** Comparison of the two precipitates (analytical data) and MAP (theoretical), based on dry matter. First precipitate includes suspended solids, while the second was produced after addition of MgO and contained 66% of the soluble P

	TOC g/kg	Ash kg/kg	Mg/P kg/kg	Ca/P kg/kg	Fe/P kg/kg	N/P kg/kg	Cd/P mg/kg
First precipitate	92	0.67	0.85	3.4	1.4	0.37	7.1
Second precipitate	1.8	0.75	0.71	0.19	0.03	0.38	0.35
MAP	0	1?	0.77	0	0	0.45	0

**Table 5** Products recovered with different methods, % of total

Method	N	P	K	VRF	Comments
<b>Urine</b> , RO	95	90	99	5	High salt, all impurities
Evaporation	95	100	99	20	Very high salt, all impurities
MAP + NH <sub>3</sub>	3 + 85	90 + 0	0 + 0	125 + 10	Relatively pure
<b>Reject</b> , RO	90	92	97	20	High salt, all impurities
Evaporation	95	100	100	50	Very high salt, all impurities
MAP + NH <sub>3</sub>	5 + 85	65 + 0	0 + 0	330 + 10	Relatively pure

**Table 6** Main resources used and estimated costs per m<sup>3</sup> treated media

Method	Chemicals	Energy, kWh		Cost at 10 m <sup>3</sup> /h, SEK		Total SEK at	
		Electr.	Heat	Capital	Operation	10 m <sup>3</sup> /h	2 m <sup>3</sup> /h
<b>Urine</b> , RO	6 kg H <sub>2</sub> SO <sub>4</sub>	8	4	6.6	16	23	32
Evaporation	13 kg H <sub>2</sub> SO <sub>4</sub>	30	0	20	28	48	78
MAP + NH <sub>3</sub>	0.6 kg MgO	0.6	66	1.9 + 3.8	2.7 + 14	22	42
<b>Reject</b> , RO	2.9 kg H <sub>2</sub> SO <sub>4</sub>	5	0	4.5	8.7	13	21
Evaporation	4.2 kg H <sub>2</sub> SO <sub>4</sub>	30	0	20	19	39	69
MAP + NH <sub>3</sub>	0.14 kg MgO	0.9	26	2.1 + 3.8	1.5 + 6.0	13	32

yield of nutrients will be a little lower. Another possibility is to distill ammonia directly from the urine, and after aeration from the reject water. Most of the ammonia will be collected in the first 100 l concentrate/m<sup>3</sup> treated media. The residue will be evaporated without addition of acid. The cost for this is estimated to 40 SEK/m<sup>3</sup> in both the urine and the reject water case. There are two main uncertainties in the discussed methods and cost estimates. The first one is the precipitation of MAP. All other separation methods are well known and tested, while the precipitation of MAP needs to be further investigated in full scale. The other uncertainty is the composition and behaviour of a real BioP reject.

## Conclusions

Although the figures are relatively rough, the following conclusions can be drawn.

Of the tested methods, concentration with reverse osmosis gives the lowest cost, while precipitation of MAP and distillation of ammonia give the products with lowest amount of contaminants.

These costs for recovered nutrients are realistic or even competitive regarding the urine because of the great amounts of nutrients recovered, but not for the case of reject water.

For urine the savings in transport costs to the farmers and in storage volume should be added, but still the high cost is the separation of urine from the rest of the sewage. It is this separation that drastically decreases the load of N and P into the STP. Source separated urine has a positive value from the start.

Reject water has a negative value, since it is just a load on the STP. In this case the savings in the STP are an important benefit caused by the separation.

A separation of P is necessary if you want to digest BioP sludge and utilise the energy in it.

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