Ammonium removal from anaerobic digester effluent by ion exchange
T. Wirthensohn, F. Waeger, L. Jelinek and W. Fuchs

ABSTRACT

The effluent of a 500 kW biogas plant is treated with a solid separation, a micro filtration and a reverse osmosis to achieve nutrient recovery and an effluent quality which should meet disposal quality into public water bodies. After the reverse osmosis, the ammonium concentration is still high (NH$_4$-N = 467 mg/l), amongst other cations (K$^+$ = 85 mg/l; Na$^+$ = 67 mg/l; Mg$^{2+}$ = 0.74 mg/l; Ca$^{2+}$ = 1.79 mg/l). The aim of this study was to remove this ammonium by ion exchange. Acidic gel cation exchange resins and clinoptilolite were tested in column experiments to evaluate their capacity, flow rates and pH. Amberjet 1,500 H was the most efficient resin, 57 BV of the substrate could be treated, 1.97 mol NH$_4$-N/l resin were removed. The ammonium removal was more than 99% and the quality of the effluent was very satisfactory (NH$_4$-N, 2 mg/l). The breakthrough of the observed parameters happened suddenly, the order was sodium—pH—ammonium—potassium. The sharp increase of the pH facilitates the online control, while the change in conductivity is less significant. A regeneration with 3 bed volumes of 2 M HCl recovered 91.7% of the original cation exchange capacity.

Key words | ammonium, anaerobic digester effluent, clinoptilolite, ion exchange

INTRODUCTION

Biogas has become an increasingly important source of alternative energy in Austria. While 119 biogas plants were operated by the end of 2003 the number almost doubled to 231 by the end of 2005 (Energie-control 2006). Not only the number, but also the size of the plants increased, leading to massive amounts of digester effluent, being still rich in organics and also in nutrients. The concentrations of nitrogen, mainly present in the form of ammonia, can be more than 100fold higher than in municipal waste water. The disposal of these residues has become the bottle neck of this sustainable energy source. Traditionally the digestate was used for manuring. However, due to the legal limits for N-fertilization per area of farmland (DüngemittelVO 1994—Austrian Law for Fertilizer Output), imposed to protect the groundwater, increasingly big areas are required and rising transport costs would emerge. An alternative option is to separate the solid residues from the liquid fraction that contains the mayor amount of the nitrogen. The less voluminous solids are again used for fertilizing whereas the liquid fraction has to undergo further treatment.

Nitrogen in water bodies promotes eutrophication (Thornton et al. 2007), ammonium causes oxygen demand (Jørgensen & Weatherley 2003). Ammonia toxicity to fish and other aquatic animals is very significant and concentrations in the range 0.2–0.5 mg/l can be fatal (Wiesmann 1994). Hence, the removal of ammonium from effluents is of great importance in the control of nitrogen pollution (Wen et al. 2006) and increasingly strict legal limits have been implemented. The traditional way for nitrogen removal from wastewater or liquid wastes is the biological process of nitrification/denitrification. (Kelly 1996; Karadag et al. 2007). But the high ammonium concentrations in anaerobic digester effluent may be inhibitory to
the biological process and also shock loads can cause trouble to biological treatment systems. Therefore, current research focuses on physical nitrogen removal methods as economically and environmentally friendly treatment alternative.

At the biogas plant investigated herein, a treatment setup consisting of a solid separation, a micro filtration and a reverse osmosis (RO) was implemented. One goal of this treatment process is the recovery of nutrients. Phosphorus, nitrogen and potassium should be extracted in a way, that it can be directly used for manuring or allows the production of a fertilizer. The second goal is the purification up to an effluent quality which meets the requirements for internal usages such as process water, irrigation on farmland or even disposal into public water bodies. In this context, anaerobic digester effluent proved to be a challenging substrate for purification (Novak et al. 2003), especially the ammonium concentration was still too high after RO treatment.

The scope of this study was to evaluate the efficiency of an ion exchange unit for final ammonium removal to meet the required limits. Within the last decades, much research has been done about the cation exchange capability of zeolite, a naturally occurring alumino silicate. Among these, clinoptilolite is considered most suitable for ammonium exchange regarding selectivity and capacity (Thornton et al. 2007). Therefore clinoptilolite was compared to polymeric ion exchange resins, since they have higher cation exchange capacity, higher reaction rates and chemical resistivity (Farkas et al. 2005).

**METHODS**

**Origin of the substrate (reverse osmosis permeate)**

A 500 kW biogas plant in Austria is operated in co-digestion mode with cow manure (50%), kitchen and bio waste (25%), slaughter waste (15%), and energy crops (10%) as a substrate. The plant produces approximately 3 m³ effluent per hour. Solids are separated with a screw press (mesh size = 1 mm) and a vibration sieve (mesh size = 0.25 mm). This pre-treatment is followed by a submerged micro filtration (MF; Mitsubishi, pore size = 0.5 μm) and a two-stage reverse osmosis (RO) for sea water desalination (Filmtec SW 30 HR 380), producing 2 m³ RO permeate per hour (Figure 1). This treatment of residues shows high reduction rates for all parameters. The micro filtration completely removes the suspended solids and most of the colloids. Therefore the COD is largely cut down in this process step. Naturally MF does not change the ammonium concentration, but it gets significantly reduced subsequently in the RO. Nevertheless, the RO
effluent is still very high. This was on the one hand due to the very high inflow concentration (NH4-N = 5,000 mg/l), on the other hand, because the treatment of anaerobic digester residuals is a fairly new topic, and the pilot plant herein was facing operational problems several times. To evaluate the capacity of the ion exchange resins, challenging conditions were applied and the experiments were conducted with an ammonium concentration of NH4-N = 467.4 ± 33.2 mg/l (Table 1 and Table 2). This concentration is significantly higher than in comparable investigations with simulated municipal wastewater, in which NH4-N ranged from 19–176 mg/l (Leakovic et al. 2000; Jorgensen & Weatherley 2003; Weatherley & Miladinovic 2004). Ion exchange, as a further process step of the effluent treatment, should help to fulfill the requirements for internal use or disposal into public water bodies.

**Description of the ion exchange process and investigations**

**Set up**

The experiments conducted addressed several aspects: in a first approach, for all ion exchange materials the cation exchange capacity (CEC) in particular for ammonium was investigated. The further experiments then focused on the most efficient ion exchanger material:

- The impact of a feed solution with a decreased pH was evaluated;
- The flow rate was tripled, therefore the contact time significantly reduced;
- The necessary amount of acid for regeneration was specified.

During these experiments, the sustainability of the CEC was observed, whether an exhaustion of the absorption capacity could be observed.

For the experiments, glass columns of an internal diameter of 1.3 cm and height of 40 cm were employed and loaded with ion exchanger beads or clinoptilolite. Table 3 summarizes the characteristics of the applied ion exchange materials. The beds had a volume of 20 cm3 and a height of 15 cm. The liquid was pumped through the columns with a peristaltic pump (Kouril PCD 83.2) in the down flow mode at a volumetric flow rate of 100 ml/h, which is equivalent to 5 bed volumes (BV) per hour, and a surface hydraulic loading of 1.26 cm3/cm2/min, which complies to an empty bed contact time (EBCT) of 12 min. The EBCT and the flow rate of 1.26 cm/min applied herein are in a common range (McVeigh & Weatherley 1990; Leakovic et al. 2000). Samples were collected automatically every 12 min (Auto Sampler FCC 61). The experiments had a duration of 15 h.

**Applied resins, activation and regeneration**

For the experiments, commercially available resins were chosen according to previous experience and recommendations of the leading producers (Rohm and Haas,
Lanxess). As further material natural clinoptilolite from Slovakia was used.

The activation of ion exchangers can be critical for the selectivity and ammonium exchange capacity (Hedström 2001). As standard conditions, the dry resins and clinoptilolite were swelled with sodium chloride solution (5% NaCl) for 24 h at room temperature. For activation the resins were fed with 20 BV sodium hydroxide (1 M NaOH), afterwards with 20 BV hydrochloric acid (2 M HCl). This flushing procedure is not only replacing cations with Na\(^+\) and subsequently with H\(^+\), but it is also cleaning and swelling the resins. The standard regeneration was accomplished with 10 BV of 2 M HCl. For activation and regeneration the same flow rate was applied than during the ion exchange experiments (flow rate = 1.26 cm/min).

### Analytics

The cations were determined with atomic adsorption spectrometry (SpectrAA 220, Varian Inc). Analyses of all other parameters were done according to standard methods for the examination of water and waste water (APHA, AWWA and WEF 1998). Conductivity and pH were measured with a multi-parameter instrument (340i, WTW). The experiments were performed at 20°C. Chemicals used were of analytical grade. The 50% breakthrough of cations was determined graphically (Figure 2).

### Investigated parameters

All parameters are reported as relative values (C/C0), except pH and conductivity. Typically, as shown in Figure 2 a sharp increase of pH, Na\(^+\), NH\(_4\)-N and K\(^+\) occurred, while the increase of conductivity was less significant. Also Mg\(^{2+}\) and Ca\(^{2+}\) showed no clear breakthrough, minor fluctuations were due to longer hydraulic retention times, when the sample collector was changed. Since the concentrations of Mg\(^{2+}\) and Ca\(^{2+}\) were relatively small, and these cations and as well the conductivity had no clear correlation to the ammonium removal, these three parameters are not displayed in the further graphs. Also in the experiments of Dimova et al. (1999) a comparable water

| Table 3 | Characteristics of the applied ion exchange materials |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Name            | Exchanger type  | Matrix          | Functional group | Mean size       |
| Amberjet 1,500 H| Strongly acidic cation | Styrene divinylbenzene copolymer | Sulfonic acid | 650 ± 50 \(\mu\)m |
| Amberlite 252 H | Strongly acidic cation | Styrene divinylbenzene copolymer | Sulfonic acid | 600 – 800 \(\mu\)m |
| Lewatit S100    | Strongly acidic cation | Styrene divinylbenzene copolymer | Sulfonic acid | 600 ± 50 \(\mu\)m |
| Clinoptilolite  | Slovakian natural zeolite | Na\(^+\) Al\(_6\)Si\(_{30}\)O\(_{72}\) Crystall channels | | 63 – 315 \(\mu\)m |

**Figure 2** | Observed parameters and graphical determination of the 50% breakthrough of eg pH (with ion exchange resin Lewatit S 100).
hardness showed no significant affect. The observed order of the cation elution corresponds to the affinity to the ion exchange material, the weaker the affinity of a certain group of ions, the sooner they get eluted.

RESULTS AND DISCUSSION

Comparison of cation exchange capacities of different resins

The polymeric exchangers generally exhibited a higher CEC and a more pronounced ion breakthrough, compared to clinoptilolite. Generally, the removal of cations was very efficient with removal rates for ammonia > 99%. Amberjet 1,500 H exhibits the best exchange capacity, followed by Lewatit S 100 and Amberlite 252 H (Table 4, Figure 3). Similar experiments with the side stream effluent of a fertilizer factory (NH₄-N = 176 mg/l) provided comparable results with CECs for Lewatit S 4428 from 1.9 meq/ml to 2.8 meq/ml (Leakovic et al. 2000). The CECs achieved in the experiments reported here exceeded the producer data slightly, presumably because the total cation concentration applied was relatively high. The total exchange capacity includes not only ammonium, but also other ions competing for exchange (Chen et al. 2002). The strongest affect has K⁺ because of its higher affinity to the exchanger (Farkas et al. 2005). Similar results are reported by Wang et al. (2007) who tested the influence of several cations on ammonium removal. The low mobility of K⁺ was also observed in the experiments.

Table 4 | Ammonium exchange capacity of the tested resins

<table>
<thead>
<tr>
<th>Resin</th>
<th>Cation exchange capacity [meq/ml]</th>
<th>Capacity according to product information [meq/ml]</th>
<th>NH₄-N exchange capacity [meq/ml]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Amberjet 1,500 H</td>
<td>2.28</td>
<td>&gt; 2.0</td>
<td>1.97</td>
</tr>
<tr>
<td>Lewatit S 100</td>
<td>2.03</td>
<td>&gt; 2.0</td>
<td>1.76</td>
</tr>
<tr>
<td>Amberlite 252 H</td>
<td>1.89</td>
<td>&gt; 1.7</td>
<td>1.64</td>
</tr>
<tr>
<td>Clinoptilolite</td>
<td>0.97</td>
<td>–</td>
<td>0.84</td>
</tr>
</tbody>
</table>

Figure 3 | Typical experiment results of Amberjet 1,500 H, Amberlite 252 H, Lewatit S100 and Clinoptilolite.
conducted here. The concentration exhibits a slow increase and reaches the initial concentration about 20 BV later than sodium. An effluent concentration of sodium, higher than the influent concentration, also suggests, that potassium with its higher affinity replaces already adsorbed sodium. Generally the adsorption of ions other than ammonium is significant and therefore will reduce the effective uptake capacity for ammonia ion and thus the economics of the process.

Beside cations, also organic contaminants are considered to reduce the ammonium uptake, as experienced in other studies (Farkas et al. 2005). Even though the substrate used for these experiments is a colorless liquid after treatment by reverse osmosis, there is still a COD of 95 ± /− 12 mg/l, due to small organic molecules, which might have a negative impact on the CEC.

Clinoptilolite has been found very effective for removing ammonia (Karadag et al. 2007), since it has a high affinity towards monovalent cations (Vokacova et al. 1986; Farkas et al. 2005; Wang et al. 2007). Still the CEC of clinoptilolite obtained in these experiments is significantly lower than for artificial resins with 0.97 meq/ml. Literature data mentions CEC values for clinoptilolite in a wide range from 0.05 – 1.15 meq/ml (Thornton et al. 2007). Generally an exchange capacity of only 20 – 25% compared to strong acidic cation exchangers is predicted. A high selectivity was observed, with the order of preference being Mg$^{2+}$ < Na$^+$ < Ca$^{2+}$ < NH$_4^+$ < K$^+$. Different to polymeric resins, clinoptilolite has a higher selectivity for monovalent ions. Wang et al. (2007) found a similar order. The order of monovalent ions of these experiments, H$^+$ < Na$^+$ < NH$_4^+$ < K$^+$, is in accordance with their findings.

Order of ion breakthrough

Most studies concerning cation preference were focusing on bivalent ions, while experiments on monovalent ions are not so common. Standard works about polymeric strong acidic cation exchangers (Helferich 1959; Inczédý 1964) suggest an affinity order of H$^+$ < Li$^+$ < Na$^+$ < NH$_4^+$ < K$^+$ < Rb$^+$ < Cs$^+$. Monovalent ions have weaker affinities than bivalent ions. Within the bivalents ions it generally applies, the smaller the cations, the stronger the affinities. In these experiments for Lewatit S 100 the sequence was H$^+$ < Na$^+$ < NH$_4^+$ < K$^+$, which is in accordance with the order mentioned above. Amberjet 1,500 H and Amberlite 252 H have a different breakthrough order. The pH is the second parameter to increase after the sodium concentration. The increase of pH shows the exhaust of the protons as exchange ions, hence in that case sodium has a lower affinity to the ion exchangers than protons and the order of affinity was Na$^+$ < H$^+$ < NH$_4^+$ < K$^+$.

Amberlite 252 H exhibits the fastest ion breakthroughs in all experiments, the slope of the observed parameters increases almost twice as fast as for Amberjet 1,500 H. For Lewatit S 100, all parameters increase with similar slope, while for Amberjet 1,500 H and Amberlite 252 H the increase of potassium is significantly more gentle. The reason for that is presumably that potassium with its higher affinity partly substitutes other cations.

For clinoptilolite, the increase of all parameters is significantly less steep. This reconfirms, that clinoptilolite needs a longer contact time of 15 – 60 min to reach equilibrium, depending on the ammonium concentration, as stated by Thornton et al. (2007) and that the current flow rates are too high for a full exchange. Therefore clinoptilolite seems more applicable for municipal waste water with its lower ammonium concentration (Vokacova et al. 1986), than for anaerobic digester effluent. In experiments with high ion concentrations Farkas et al. (2005) determined the ion selectivity of clinoptilolite. A high selectivity was observed, with the order of preference being Mg$^{2+}$ < Na$^+$ < Ca$^{2+}$ < NH$_4^+$ < K$^+$. Different to polymeric resins, clinoptilolite has a higher selectivity for monovalent ions. Wang et al. (2007) found a similar order. The order of monovalent ions of these experiments, H$^+$ < Na$^+$ < NH$_4^+$ < K$^+$, is in accordance with their findings.

Factors influencing the ammonium adsorption

Feed with decreased pH

Within the tested ion exchangers, Amberjet 1,500 H proved to be the resin with the highest CEC. Further experiments were conducted with this resin to test the impact of a pH decrease in the feed solution. Ammonia-ammonium equilibrium in solution is largely pH dependant (Hedström 2001) and it stands to reason that only the ionised from can be removed from solution by ion exchange
At the current conditions of pH 8.05, 95.7% is present as ammonium according to Equation (1) (Ammonia/Ammonium balance).

\[
\frac{[\text{NH}_3]}{[\text{C}_138]} = \frac{[\text{NH}_3 + \text{NH}_4^+]}{1 + \frac{1 + 10^{pK_a-pH}}{1 + 10^{pK_a-pH}}}.
\]

\[pK_a = 4 \times 10^{-8} \times T^3 + 9 \times 10^{-5} \times T^{2-0.0356} \times T + 10.072\]

0.1 M HCl was used to lower the pH from 8.05 to 5. As a result, a significant increase of the CEC by 27% was observed. This is in contrast to other experiments where only little effect in the range of pH 5 and 8 (Karadag et al. 2007) is reported. In other studies the optimum pH for ammonium removal is pH 6–7 (Sheng & Chang 1996; Chen et al. 2002). Below pH 5 a significant reduction of the CEC is expected due to the higher hydrogen ion concentration, which causes competition for sites, resulting in a reduction of the performance. A higher pH in the effluent is due to protons replacing already adsorbed sodium, which also causes the high Na\(^+\) concentration in the effluent.

**Contact time**

Another experiment addressed the evaluation of the contact time for ion exchange (Figure 4b). While contact times in batch experiments range up to 5 d to reach equilibrium (Weatherley & Miladinovic 2004), the empty bed contact times (EBCTs) in dynamic column experiments are in the range from 1.5 to 15 min. After a standard EBCT of 12 min in the experiments described above, the flow rate of 1.26 cm/min was tripled to 3.77 cm/min (≈15 BV/h) resulting in an EBCT of 4 min. This enhanced flow rate had no significant impact on the CEC, only the elution of all different cations occurred at nearly the same time—no distinct sequence of the single breakthrough curves could be observed. Also the slope of the ion breakthrough was less steep, indicating that towards the end of the capacity the reduced contact time was insufficient for full adsorption. These results coincide with the reports of other authors (Semmens et al. 1977), who claim that the shapes of the breakthrough curves depend on the flow rate. In major scale experiments even flow rates of 40 BV/h were successfully applied to treat effluent of a fertilizer factory (Leakovic et al. 2000). However, this was only possible because of the lower NH\(_4\)-N concentration of 176 mg/l but also underlines the high cation-exchange capability and quick reaction rate of polymeric resins (Farkas et al. 2005).

**Exhaustion of the adsorption capacity**

After 10 circles under standard conditions the CEC did not show a significant decrease. Ji et al. (2007) observe a 0.2% decrease of CEC after 5 cycles, but most experiments are done over a longer period. Ancuta et al. state 99.8%–97.0% CEC after 100 cycles, also Lahav & Green (1998) observe a constant CEC.

**Regeneration of resins and long term stability**

Both upflow and downflow applications were tested in other lab scale experiments for regeneration (Hedström 2003). The downflow mode seems more practical and therefore was used in these tests. For scientific purposes, e.g. to evaluate the sorption capacity, often a 10fold stoichiometric ratio of cations is applied to ensure complete regeneration. In opposition to that, in practical
applications, the amount of regeneration chemicals is kept as low as possible, since regeneration can make up to 80% of the process costs (Lahav & Green 1998; Dimova et al. 1999). In general, the higher the concentration of the ions in the eluate and the smaller the volume of the eluate, the higher the regeneration efficiency of the regenerant. The usage of a highly concentrated regenerant in combination with a long EBCT can help to keep the amount of washing brine as small as possible. Still certain limits have to be obeyed. A certain flow rate is required to transport the ions out of the column, otherwise an equilibrium forms between the washing solution and the resin. Furthermore, highly concentrated acids for regeneration can damage the resin beads. E.g. Leakovic et al. (2000) used highly concentrated nitric acid of (56 wt.%) for regeneration, but this is not recommended by the resin producers. The effect of concentrated hydrochloric acid is much milder since it has no oxidative properties.

In the investigated case, the regeneration with 5 BV of 2M HCl (10 meq/ml) at a flow rate of 1.26 cm/min achieved a full regeneration. This amount of regeneration brine is equivalent to a 5fold stoichiometric ratio. Ancuta et al. (2005) use only 0.7 BV of a 20% HNO₃ (2.45 meq/ml) at a flow rate of 0.13 cm/min for the resin Amberlite 252. The back draw of this small employment of chemicals is the lower degree of regeneration between 36.9% and 47.6%. Some studies consider this the normal operation range: in practical applications it is difficult to obtain an operating exchange capacity that exceeds 50% of CEC. According to information provided by the producers of the resins 60% recovery is recommended as the typical operation range.

The order of the elution of cations was similar to the order of the breakthrough after adsorption, ammonium being eluted first, followed by sodium and potassium with their stronger affinities. The elution of ammonium has the concentration peak from 1.5 to 2.5 BV washing solution, at 5 BV all ammonium is eluted, while it takes almost 10 BV, before all the potassium is eluted. To minimize acid requirement, a regeneration was performed with 3 BV. In the following experiment exhibited a slightly reduced CEC of 2.12 meq/ml (91.7%) (Figure 5). This shows, that 3 BV are sufficient to elute ammonium and also most of the potassium and sodium cations. For cost considerations, the regeneration with 3 BV is more recommendable. In such a case an enhanced regeneration is necessary after a couple of circles to remove Mg²⁺ and Ca²⁺. Even if they are present only at minor concentrations, they have a much higher sorption affinity than the monovalent ions (Chen et al. 2002). Also other literature data suggests, that calcium ions are strongly sorbed to the exchanger and permanently decrease its ion exchange capacity (Vokacova et al. 1986; Hedström 2001) and therefore need to be removed from time to time.

Recommendations for a practical application

The results of the laboratory scale experiments served as the basis for the design of a full scale plant at the given site. A downflow mode is recommended as it requires less pumping effort. To keep costs for regeneration low, a regeneration level of 60% was chosen. For the treatment of 2 m³/h RO permeate, an ion exchanger volume of 850 l is needed, to perform regeneration every 12 h. Instead of
hydrochloric acid the use of sulfuric acid is suggested, which is not only less costly but even provides two protons per molecule. Approx. 100 l of sulfuric acid (50% w/w) are calculated for regeneration. The high concentration will help to keep the amount of washing brine small. In certain applications calcium sulfate precipitations caused problems but that is not expected at the low calcium concentrations herein.

Practical tests have demonstrated that the residual washing brine can be recycled to the reverse osmosis. Sulfate, as a big anion, is retained by the RO membrane, and therefore also an equal amount of cations will be removed (Hochstrasser 2007). The RO concentrate will be enriched with nitrogen and sulfur, which makes it more precious as fertilizer. As final usage, the RO concentrate can be dewatered into a dry fertilizer. Surplus heat is available from the combined heat and power station of the biogas plant. Alternatively, the RO concentrate can also be used as liquid fertilizer. Since it is very concentrated, less transport costs arise.

Even though the preceding RO removes all particles, still precipitates can form and lead to cloggings or hydraulic short cuts which can significantly decrease the efficiency of the column. During the experiments, cloggings were also caused by gas bubbles. They can occur when the low pH in the ion exchange column releases CO₂ or adsorbed NH₄⁺ turns to gaseous NH₃, when the pH rises after the CEC is exhausted. In any case, to remove cloggings, facilities for back flushing should be provided.

CONCLUSIONS

Ion exchange proved to be a successful and stable process step to upgrade the anaerobic digestate treatment and the achieved results provided valuable data for the design of a full scale plant. The legal limits for disposal into public water bodies could be met by both the polymeric strongly acidic resins and clinoptilolite, while the nutrients were recovered in the washing brine. Due to higher exchange capacities, reactivities and long durabilities, the polymeric resins are more recommendable than clinoptilolite. Ammonium removals of more than 99% at contact times of 12 min were achieved. For a more economical application, the flow rate can even be increased. The regeneration with acid containing a 3 fold stoichiometric ratio of protons is sufficient to maintain the cation exchange capacity.

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