The soil purification capacity for treating pig slurry was evaluated over five years using a hydrologically isolated field treatment plant, the so-called “Solepur” process. This involves three operations: (1) overdosing the managed field with surplus slurry, (2) collecting and treating the nitrate rich leachate, (3) irrigating the final treated water over other fields. The facility consists of (1) a managed field (3280 m²) which allows the total recovery of all the leachate water which percolates through growing ryegrass, Lolium perenne, to which the pig slurry is applied, (2) a storage-pump-reactor system for denitrification and (3) a non-managed field for completing treatment.

The purpose of this study was to evaluate over a five year monitoring period, the feasibility and performances of such a process, and to determine the optimum operating conditions. From 1991 to 1995, 4931 m³/ha of raw pig slurry was applied to the managed field, which represents a nominal load of 986 m³ ha⁻¹ yr⁻¹. This process decreased the COD of pig slurry by 99.9% and removed 99.9% of phosphorus and approximately 90% of nitrogen. The Solepur’s final product is a limpid leachate containing a very low concentration of organic matter, but with relatively high nitrate levels resulting from oxidation of slurry nitrogen in soil. The extent of nitrification efficiency was satisfactorily demonstrated both through soil inorganic-N profiles and through continuous analysis of the leachate water leaving the overdosed field which shows nitrate concentration up to 1500 mg/l. Subsequently, the nitrogen removal from the nitrate rich leachate was achieved through batch denitrification sequences: raw pig slurry (which supplies carbon to support biological denitrification) is added to a volume of 150 to 250 m³ of drainage water (representing a nitrogen load of 10 to 40 kg N) in the ratio 1:20 to 1:100 v/v. Parallel laboratory tests showed the influence of C/NO₃⁻-N ratio on the efficiency of nitrate nitrogen removal and demonstrated the feasibility of semi-continuous denitrification. It was confirmed that a C/NO₃⁻-N ratio of three is necessary to achieve a complete denitrification sequence. The results conclusively show that the Solepur process is a promising waste treatment system.

1. Introduction

Brittany (Western France) is well identified as one of the main regions of intensive animal production in the EC, since it is at the heart of the French pig and poultry industry in an area which produces more than 50% of the pigs, and 41% of poultry, within 7% of the agriculturally used land. This situation generally creates localized areas with the nitrogen load in excess of the farm’s nitrogen requirements. Slurries comprise a large potential source of nitrate in ground water. The safe and efficient disposal of farm wastes, particularly pig slurry, is becoming a subject of widespread concern in most areas of specialized livestock production. Control of water pollution, especially by nitrates and increasingly phosphates, is highlighted in the array of existing and proposed legislation (EC Directive 91/676/EEC). Therefore, there is a requirement for new, cost effective techniques, which will reduce the pollution potential from slurry on pig farms.

Biological treatment remains one of the most popular methods available for controlling pollution from livestock wastes. Pig slurry contains high concentrations of organic matter, nutrients and microorganisms. The main options for removing this byproduct are (1) conventional treatment methods, (2) agronomic utilization through land spreading and, (3) natural recycling through adapted landscapes and aquatic systems. Conventional methods such as anaerobic digestion and aerobic treatment can be used to reduce offensive odours from pig slurry and to remove nitrogen and...
carbon.\textsuperscript{7-9} Alternatively, sedimentation techniques were shown to have the potential to concentrate the insoluble matter of treated slurry into a sludge phase, thus complementing aerobic processes.\textsuperscript{10} These conventional processes and further the central processing units require sophisticated equipment and skillful operation.\textsuperscript{11}

Spreading on land is the current agricultural practice but on most pig farms, there is a lack of land available to dispose of the slurries produced. It is now regulated by restrictive legislation, to avoid pollution problems from excess nitrates in surface waters and aquifers.

As an alternative to land spreading, some investigators have suggested using simple methods through natural soil and water processes to treat pig slurries.\textsuperscript{12} Polprasert \textit{et al.}\textsuperscript{13} evaluated the treatment performance of water hyacinth ponds and observed percentage removal efficiencies of 50 to 90\% for carbon and total nitrogen from a pig wastewater influent.

The soil is a well-known purifying system.\textsuperscript{14,15} It has been used successfully to treat a variety of domestic and industrial waters,\textsuperscript{16,17} and pig and cattle wastes.\textsuperscript{18-20}

The soil treatment process developed in the USA and called a barriered landscape wastewater renovation system (BLWRS) consists of a mound of soil underlain by an impermeable barrier and drainage system which creates an aerobic zone in the top of the BLWRS and an anaerobic zone next to the impermeable barrier (Ritter and Eastburn\textsuperscript{19}).

The system developed in France by Bertrand (reported by Bertrand \textit{et al.}\textsuperscript{21,22}) and identified as "Solepur" involves reserving a field which is managed to take surplus slurry. It is based on a BLWRS approach, except that the drainage water collected from the field (nitrate rich), is subsequently denitrified in an open basin. Denitrification requires the availability of an electron donor and the organic matter present in pig wastes is able to satisfy this need. The denitrified liquid is then recycled back to the field through irrigation.

The initial stage of this work was a preliminary laboratory study to test the approach. The extent of nitrification was evaluated in experiments using monolith lysimeters, and denitrification was studied during separate laboratory experiments.\textsuperscript{21} Once this initial work had been carried out, it was decided to evaluate this approach further using a field treatment plant.\textsuperscript{23} This paper reports:

1. a description of the Solepur field treatment plant and its main characteristics;
2. the soil purification performances; and
3. the nitrogen removal processes including denitrification performed \textit{in situ} and during laboratory studies.

2. Methods and procedures

2.1. The Solepur treatment process

The Solepur treatment unit (Fig. 1) consists of (1) a drained and hydrologically isolated field of 3280 m\textsuperscript{2} which allows the total recovery of all the leachate water which percolates through growing ryegrass, \textit{Lolium perenne}, to which pig slurry is applied; (2) a system of storage-pump-reactor for denitrification and (3) a non-managed field for completing treatment. The process involves the following operations:

1. overdosing the managed field with surplus slurry,
2. collecting and treating the nitrate-rich leachate,
3. irrigating the final treated water over other fields.

The first operation of slurry spreading takes place during spring and summer, while the second operation of drainage water denitrification is mainly performed during autumn and winter.

The unit has been operating since December 1990. It was installed in spring 1990 on a structured and well-drained silty soil (soil texture: 14.1\% clay; 22.9\% sand; 63.4\% silt) with 182% total carbon, 0.178% total nitrogen and an initial pH of 5.6.

2.1.1. Pilot facility design and construction

The initial stage was the design of the drainage system using a "laser" equipped machine which locates the position of the drains, before installing them.

Two main layers were distinguished, top soil (0–30 cm depth) and subsoil (30–80 cm depth). First the topsoil was entirely removed, then the subsoil progressively. The plot was completely isolated hydrologically by means of a "polyane" film placed at the base of the drains. The drains made of hard polyvinylchloride were then put on the film at previously selected places. To avoid water stagnation, each 6 m length was installed with a slope, the drain being placed down the middle of the slope. The drains were then filled in by gravel and the gravel was covered by an additional plastic film ("Bidim", a permeable antifouling membrane) in order to avoid root colonisation and blocking of the drains. The subsoil, from which stones and pebbles were separated, was then replaced on the Bidim film. A control plot of 100 m\textsuperscript{2} was constructed in a similar way.

Six drains of 74 m length and 100 mm diameter, each 6 m apart, were necessary in the main plot, of
area 3280 m² (82 m length × 40 m width). Two drains of 10 m length and 100 mm diameter were used in the control plot of area 100 m² (10 m × 10 m). The drainage water collected by the 100 mm diameter PVC tubes, passes gravimetrically to the field laboratory and then to the tanks which form the second part of the treatment facility.

Three plastic lined lagoons were installed as shown in Fig. 1. The intermediate storage tank (450 m³ capacity) received the drained water, which was sub-

![Fig. 1. The Solepur field treatment process](image)

![Fig. 2. Pilot plant instrumentation](image)
sequently transferred to the denitrification reactor tank (350 m$^3$ capacity). There was also the possibility of moving the drained water directly to the denitrification reactor. A slurry storage lagoon (150 m$^2$ capacity) allowed the slurry to be pumped and spread on the plot.

A special slurry pump (Model AFI L20, DODA Ltd, Canicossa, Mantova, Italy) of 60 m$^3$/h capacity was used to bring the slurry to the plot by means of an irrigation machine (Model 1R2, Ocmis Irrigazione Ltd, Castelvetro, Italy) equipped with a spraying boom of 38 m width. This allowed uniform slurry distribution on the plot.

2.1.2. Pilot facility instrumentation

Instruments installed (Fig. 2) in the field are an ultrasonic flow meter (Model 3210, Isco Inc., Lincoln, USA), a wastewater sampler (Model 2100, Isco Inc., Lincoln, USA) connected to the flow meter and three probes (Ponselle Ltd, Virolay, France) including pH, conductivity, redox and temperature. The data was automatically recorded on an electronic datalogger (SAB 600 AGM, CR2M Ltd, Massy, France) and transferred to a portable computer before being processed at the research centre of Cemagref-Rennes.

2.1.3. Pilot facility measurements and monitoring

2.1.3.1. Slurry sampling. The slurry used came from the commercial farm adjacent to the Solepur pilot process. The first two slurry applications were performed during winter months with rainwater diluting the slurry pit. Thereafter, it was decided to have no slurry applications during winter, because of adverse soil conditions, and to fill the storage tank just a few days before the date fixed for slurry application, in order to apply raw slurry.

The raw pig slurry was applied to the managed field in doses each month from March/April to October/November each year, depending on the suitability of the soil and on weather conditions. During each day, slurry application was split into two or three spreadsings and a sub-sample of 2 to 3 l was regularly taken during the spray boom operation. At the end of the spreading, the total sub-samples represented a volume of 40 to 50 l of slurry. The collected slurry was then gently stirred and two samples of 11 each were taken to perform full chemical analyses. So, each batch of slurry applied to the field was analysed and 52 batch analyses were performed during the five years.

During 1994, four intensive pig slurry applications were performed on bare soil on 10 May; 8 June; 24 June and 20 July. The soil was ploughed one or two days after each slurry application to allow homogeneous soil sampling.

2.1.3.2. Drainage water sampling. During the whole drainage season, usually starting by the end of September, a daily sample of drainage water was taken by means of the automatic sampler located in the field laboratory. This 1 l daily sample was composed of four 250 ml subsamples collected at 6 h intervals. All of the daily samples were analysed for their nitrate content. Furthermore, a composite sample was prepared each week for a full chemical analysis.

2.1.3.3. Denitrification effluent sampling. At the start of an experiment, 150 to 250 m$^3$ of the leachate from the intermediate storage tank was pumped into the denitrification reactor and mixed with 3 to 8 m$^3$ of raw pig slurry. This mixture was left undisturbed to denitrify for various period of time. Then the denitrified effluent was discharged for irrigation while a small fraction of the total volume was left in the denitrification reactor as inoculum for the next batch. From previous laboratory studies, it was assumed that with raw pig slurry added as an organic material, an empirical carbon to nitrogen ratio of 3:1 was necessary to support complete denitrification. In our study, variable amounts of slurry were used during the batch cycles to deliberately manipulate the C/N ratio up to 25:1.

Effluent sampling in the reactor tank, at different intervals during the course of denitrification, was initially accomplished by a portable manual pump (Isco, model PTP-100). A systematic sampling protocol was applied at each sampling time (eight sampling points within the surface of the basin taken at depths of 1 m and 2 m). This reliable but labour-consuming sampling design was used for two years, after which the pump installed to irrigate the denitrified effluent to the final treatment plot was adapted so that the effluent could be recirculated within the tank. A tap allowed regular interception and sampling of the effluent during recirculation. Usually, the batch denitrification was monitored weekly.

2.1.3.4. Soil sampling. The extent of nitrification was determined by soil sampling at different dates, March 1991; October 1991; March 1992; April, May, June, July and August 1994. The main plot was divided into four equal parts and a microplot of 1 m$^2$ was examined in each part. Nine core samples of soil were taken from each microplot and all the samples were thoroughly mixed by sieving through a 6 mm mesh. After mixing, an average soil sample of about 1 kg of fresh soil was taken, and the remaining soil replaced in the appropriate hole. Three layers were sampled (0–20, 20–40, 40–60 cm depth). Because the drainage system was only 80 cm deep, the last layer
(60–80 cm depth) was not sampled, to avoid disrupting the watertightness of the plot. During the fourth campaign of slurry applications (1994), the build up of nitrates in the soil was determined after each application on the 0–30 cm depth layer. Fresh soil was extracted for 1 h with 1 mol KCl solution (1:3 (w/v) soil solution) and filtered through Whatman No. 42 filter paper. Inorganic-N (NH$_4^+$-N and NO$_3^-$-N) was measured in the KCl extract by distillation of an aliquot with MgO and Devarda’s alloy.

2.2. Parallel laboratory trials

The bench-scale treatment system consisted of a series of denitrification reactors of 60 l capacity (polyethylene cylinders) and a peristaltic pump. The drainage water used during all laboratory studies came from the field experiment. Two separate experiments were carried out.

2.2.1. Batch denitrification

The object of this first trial was to study the influence of the C/N ratio on the efficiency of denitrification. Each of the four reactors contained 50 l of drainage water with a nominal nitrate concentration of 500 mg/l. Four volumes of slurry, nil, 0·4 l, 2 l and 6 l, was then added to each reactor to reach C/N ratios of 0, 0·5, 2·5 and 8·2 respectively. After appropriate intervals, a few ml of sample was removed to determine the extent of denitrification.

2.2.2. Semi-continuous denitrification

The object of this second trial was to evaluate during a semi-continuous study the denitrification potential remaining in a mixed liquor from the previous batch study i.e. with different levels of carbon available. An equal volume (15 l) of the mixed liquor from the four treatments collected at the end of the previous experiment was continuously fed with a single solution of drainage water with a nominal nitrate concentration of 500 mg/l. The flow rate was kept at 100 ml/h. Again, after appropriate intervals, a few ml of sample was removed to determine the extent of denitrification.

2.3. Laboratory analyses

Each batch of slurry spread on the top of the managed field was analysed using current laboratory methods for total solids (TS), total Kjeldahl nitrogen (TKN), total ammoniacal nitrogen (TAN), total carbon, chemical oxygen demand (COD), total phosphorus, total potassium and the metals copper and zinc as described in Standard Methods for the Examination of Water and Wastewater.

Weekly samples of drainage water were analysed for chemical oxygen demand (COD), biochemical oxygen demand (BOD), nitrate, nitrite, ammonia, total Kjeldahl nitrogen, total phosphorus, phosphates (PO$_4^-$), potassium (K), calcium (Ca), chloride (Cl), and sulphates (SO$_4^-$).

Mixed liquor from denitrification batches were regularly analysed for nitrite (colorimetric method), nitrate (cadmium reduction method) and total ammonia-nitrogen.

3. Results

3.1. Slurry applications

From 1991 to 1995, 4931 m$^3$/ha of raw pig slurry was applied to the managed field during 30 spreading events, which represents a nominal load of 986 m$^3$ ha$^{-1}$ yr$^{-1}$ (i.e. an hydraulic load of 99 mm). The main characteristics of the thick slurries applied (mainly from fattening pigs) are listed in Table 1. The composition of the different batches analysed from 1991 to 1995 was quite variable (52 batches analysed). The trend over the years was towards application of more concentrated slurries, which slightly reflects the new practice in pig farming in Brittany aimed at saving water. This change particularly affected total solids, which exceeded 100 kg per m$^3$, and total Kjeldahl nitrogen which was as high as 8 kg N/m$^3$ slurry during the 1994 spreading year. The increase of these two parameters over the years was close to 60%. From 1991 to 1995, the overall nutrient load was, 4950 kg/ha yr of total nitrogen, 1579 kg/ha yr of total phosphorus and 1753 kg/ha yr of total potassium respectively. On average, the rate of carbon applied during the five years of slurry spreadings was 3910 kg/ha total carbon for each slurry application. During the two last years of repeated spreadings, 1994 and 1995, 80 t/ha and 102 t/ha of COD were applied, which represents a daily nominal load ranging from 200 to 280 kg/ha d of COD.

3.2. Soil purification performances

In the Solepur process, the soil is the primary treatment stage acting as a physical and biological barrier. Its retention and biodegradation performances are mainly evaluated through scrutinized
analysis of the leachate water which percolates through the soil.

The field observations presented in Table 2, demonstrated a high efficiency of the living soil filter system which enabled the transformation of the thick viscous raw slurry to clear drainage water. In particular, the redox values indicated that aerobic conditions prevailed in the drainage water. However, the conductivity of the drainage water was high, indicating a high concentration of dissolved salts. The conductivity tended to increase from 1000–1500 μS/cm during drainage seasons 1992/1994 to 2000–2500 μS/cm during the later drainage season (1994/1995). In comparison, the values of conductivity of the drainage water from the control plot ranged from 200–300 μS/cm. The typical figures for rainwater are about 50–100 μS/cm (drinking water should have a conductivity lower than 400 μS/cm).

The results from the continuous measurements of drainage water quality are summarized in Table 3. In addition to the visual observation (clear water) quoted above, a near complete removal by the soil of the high organic load applied with the slurries was found. During the last two drainage seasons which were intensively monitored, chemical oxygen demand (COD) values lower than 50 mg/l were systematically found which, if compared with the actual COD values of the slurry influent (50 000–200 000 mg/l), indicate a 99.9% removal efficiency. The values for the biochemical oxygen demand (BOD₅) of the drainage water were near undetectable (1–5 mg/l). Additionally, dissolved organic carbon concentrations were measured during the drainage season 93/94 and were found to be about 10–15 mg/l (Lineres, personal communication).

Apart from separate soil mineral-N measurements, the nitrifying activity of the soil was also continuous as shown by the nitrate rich leachate which percolated through the soil profile. The nitrate concentration increased with increase in the nitrogen from repeated slurry applications. Peaks in nitrate concentrations of 500, 1000, and 1500 mg/l during the winters of 1992, 1993, and 1994 respectively were observed.

Significant phosphorus concentrations in the drainage water (less than 0.1 mg/l) were not found. In spite of the fact that concentrations of phosphorus in the soil solution progressively increased, particularly in the top horizon of 0–20 cm depth, from 0.062 mg P/l in 1991 up to 2 mg P/l in 1994 (Fardeau and Martinez²⁸), the strong fixing capacity of the soil retained most of the high amounts of slurry phosphorus applied. So, the soil removal efficiency for phosphorus was near 100%.

Table 1
Mean composition of the slurries used during the five years of application. (Number of batches and standard deviation in brackets)

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</thead>
<tbody>
<tr>
<td>Total solids (kg/m³)</td>
<td>54 (11, 32)</td>
<td>65.2 (8, 29.4)</td>
<td>57.5 (12, 26.1)</td>
<td>86.4 (10, 16.6)</td>
<td>67.8 (9, 23.4)</td>
</tr>
<tr>
<td>Total Kjeldahl nitrogen (N) (kg/m³)</td>
<td>4.7 (13, 1.7)</td>
<td>4.7 (8, 1.7)</td>
<td>5.1 (12, 0.9)</td>
<td>6.8 (10, 0.8)</td>
<td>5.7 (9, 0.8)</td>
</tr>
<tr>
<td>Total ammoniacal nitrogen (kg/m³)</td>
<td>2.7 (13, 0.9)</td>
<td>3.2 (8, 1.1)</td>
<td>3.5 (12, 0.5)</td>
<td>4.4 (10, 0.5)</td>
<td>3.8 (9, 0.4)</td>
</tr>
<tr>
<td>Total carbon (kg/m³)</td>
<td>23.7 (10, 12.1)</td>
<td>29.1 (8, 14.7)</td>
<td>22 (12, 10.6)</td>
<td>35.6 (10, 7.3)</td>
<td>26.0 (9, 11.7)</td>
</tr>
<tr>
<td>COD (kg/m³)</td>
<td>— —</td>
<td>— —</td>
<td>38 (2, —)</td>
<td>114 (10, 30.2)</td>
<td>98.3 (9, 68.1)</td>
</tr>
<tr>
<td>Total phosphorus (P) (kg/m³)</td>
<td>1.5 (10, 0.7)</td>
<td>1.7 (8, 0.8)</td>
<td>1.6 (12, 0.9)</td>
<td>2.2 (10, 0.4)</td>
<td>2.02 (9, 0.7)</td>
</tr>
<tr>
<td>Total potassium (K) (kg/m³)</td>
<td>1.5 (10, 0.5)</td>
<td>1.6 (8, 0.6)</td>
<td>1.8 (12, 0.3)</td>
<td>2.3 (10, 0.2)</td>
<td>1.91 (9, 0.1)</td>
</tr>
<tr>
<td>Copper (mg/kg)</td>
<td>33.6 (10, 17.2)</td>
<td>37.5 (8, 15.9)</td>
<td>42.8 (12, 18.7)</td>
<td>48.6 (10, 7.3)</td>
<td>42 (9, 11.7)</td>
</tr>
<tr>
<td>Zinc (mg/kg)</td>
<td>55.2 (10, 21.1)</td>
<td>48.7 (8, 22.3)</td>
<td>51.1 (12, 23.9)</td>
<td>65.0 (9, 11.7)</td>
<td>67 (9, 15.2)</td>
</tr>
</tbody>
</table>

— not measured.

Table 2
Field measurements of physico-chemical characteristics of drainage water. (Average values over the three years monitoring period)

<table>
<thead>
<tr>
<th>Aspect</th>
<th>Average</th>
<th>Range</th>
<th>Trend</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>4.92</td>
<td>4.24–5.46</td>
<td>decrease</td>
</tr>
<tr>
<td>Conductivity μS/cm</td>
<td>1694</td>
<td>1318–2500</td>
<td>increase</td>
</tr>
<tr>
<td>Redox mV (Calomel probe)</td>
<td>491</td>
<td>418–551</td>
<td>stable</td>
</tr>
<tr>
<td>Temperature °C</td>
<td>12.7</td>
<td>9–19.3</td>
<td>—</td>
</tr>
</tbody>
</table>
The potassium applied with the slurry is readily leachable through the drainage water. The results show a substantial increase in potassium concentration over the years with values up to 280 mg/l obtained during the last drainage season (Table 3).

3.3. Soil nitrification

Table 4 shows inorganic-N profiles determined during the first year of the experiment. We found 250 kg mineral-N/ha in March 91. This relatively high inorganic-N content was due to slurry applied in December and January being partly nitrified and partly taken up by the ryegrass growing in the plot. The second mineral-N profile was determined on the 29 of October 1991, one month after the last slurry spreading. More than 400 kg NO$_3^-$-N/ha had accumulated from March to April. An unknown amount was probably accumulated in the unsampled layer (60–80 cm) closest to the drains. The ammonium content of the soil profile at this same date was found to be low; there is apparently no ammonium accumulation in the soil. When the soil profile was examined again in March 1992, the mineral-N content was low (65 kg NH$_4^+$-N/ha and 65 kg NO$_3^-$-N/ha). It is interesting to try to follow the behaviour of this mineral-N content during the autumn/winter months. A preliminary nitrogen balance shows that the main part, 178 kg N/ha of the mineral-N content from October 1991 was lost during the autumn / winter months. A preliminary study to follow the behaviour of this mineral-N content during the autumn / winter months shows inorganic-N profiles determined during the fourth year of spreading (1941) on a bare soil. The first sampling (19/04/94) showed a low inorganic-N background of 130 kg N/ha before the new slurry spreading began. The average ammonium nitrogen application for each of these four spreading

### Table 3
Mean concentration of the Solepur field treatment plant leachate (i.e. drainage water) during four drainage seasons (range in brackets)

<table>
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<tbody>
<tr>
<td>COD (mg/l)</td>
<td>77 (60–100)</td>
<td>43 (20–75)</td>
<td>28 (20–45)</td>
<td>56 (19–110)</td>
</tr>
<tr>
<td>BOD$_5$ (mg/l)</td>
<td>5 (4–6)</td>
<td>3 (0–5–7)</td>
<td>1 (0–1–8)</td>
<td>1.4 (1–18)</td>
</tr>
<tr>
<td>NO$_3^-$ (nitrate) (mg/l)</td>
<td>290 (235–350)</td>
<td>604 (390–826)</td>
<td>886 (297–1430)</td>
<td>940 (350–1480)</td>
</tr>
<tr>
<td>NO$_2^-$ (nitrite) (mg/l)</td>
<td>0–9 (0–3–1–9)</td>
<td>0.04 (0–0–31)</td>
<td>0.05 (0–0–0–8)</td>
<td>0.09 (0–0–0–65)</td>
</tr>
<tr>
<td>NH$_4^+$ (ammonium) (mg/l)</td>
<td>0–12 (0–1–0–2)</td>
<td>0.06 (0–28–1–23)</td>
<td>1 (0–51–1–39)</td>
<td>1.2 (0–5–4–8)</td>
</tr>
<tr>
<td>TKN (mg/l)</td>
<td>10 (4–5)</td>
<td>4 (2–6–4)</td>
<td>4 (2–6–4)</td>
<td>4 (2–6–4)</td>
</tr>
<tr>
<td>PO$_4^3-$ (mg/l)</td>
<td>0–13 (0–1–0–2)</td>
<td>0.02 (0–0–1)</td>
<td>0.06 (0–5–0–1)</td>
<td>0.08 (0–5–0–5)</td>
</tr>
<tr>
<td>Total P (mg/l)</td>
<td>0–57 (0–3–0–7)</td>
<td>0.03 (0–0–2)</td>
<td>0.05 (0–2–0–6)</td>
<td>0.03 (0–0–1–9)</td>
</tr>
<tr>
<td>K (mg/l)</td>
<td>49 (31–85)</td>
<td>57 (45–69)</td>
<td>128 (94–156)</td>
<td>203 (120–280)</td>
</tr>
<tr>
<td>Ca (mg/l)</td>
<td>201 (128–340)</td>
<td>183 (120–220)</td>
<td>200 (102–300)</td>
<td>161 (90–220)</td>
</tr>
<tr>
<td>Cl (mg/l)</td>
<td>— —</td>
<td>256 (145–315)</td>
<td>174 (91–225)</td>
<td>238 (122–353)</td>
</tr>
<tr>
<td>SO$_4^2-$ (mg/l)</td>
<td>— —</td>
<td>127 (70–270)</td>
<td>175 (128–390)</td>
<td>203 (164–240)</td>
</tr>
</tbody>
</table>

---

$^a$ Batch analyses from November 1992 to April 1993 (about 750 m$^3$ drainage water analysed).

$^b$ Weekly analyses, drainage period from October 1993 to May 1994 (1200 m$^3$ drainage water analysed).

$^c$ Weekly analyses, drainage period from October 1994 to March 1995 (1500 m$^3$ drainage water analysed).

$^d$ Weekly analyses, drainage period from October 1995 to March 1996 (1000 m$^3$ drainage water analysed).

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### Table 4
Evolution of soil inorganic nitrogen content (0–60 cm depth), following the first intensive slurry application year of 1991 (standard deviation in brackets, n = 4)

<table>
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<tbody>
<tr>
<td>Ammonium (NH$_4^+$-N) kg/ha</td>
<td>138 (53)</td>
<td>67 (77)</td>
<td>64 (55)</td>
</tr>
<tr>
<td>Nitrate (NO$_3^-$-N) kg/ha</td>
<td>110 (54)</td>
<td>516 (64)</td>
<td>65 (64)</td>
</tr>
<tr>
<td>Total mineral-N kg/ha</td>
<td>248 (21)</td>
<td>583 (67)</td>
<td>129 (69)</td>
</tr>
</tbody>
</table>
was higher than 800 kg NH$_4^+$-N/ha. The behaviour of this total ammoniacal slurry nitrogen was then followed after each application, so that the nitrate build up in the soil could be monitored. The nitrification rate was then calculated as mg of NO$_3^-$-N produced divided by the time in days between two samplings. This rates varied from 5 to 14 mg NO$_3^-$-N per kg of soil/d. In spite of the high rates of ammonium applications, there was no accumulation of this form in the soil but rather a rapid oxidation to nitrate. The results presented in Fig. 3 (4 replicate microplots per sampling) were obtained within coefficients of variation (CV%) ranging from 3% to 15%. The percentage recovery of the slurry ammoniacal nitrogen applied at each application either as nitrate or remaining ammonium in the soil, was calculated and established to be 88%, 104%, 64% and 95% for the four respective applications.

### 3.4. Drainage water denitrification

#### 3.4.1. Field batch denitrification

The subsequent denitrification of the nitrate rich leachate collected from the field is the secondary treatment of the Solepur process. A number of in situ batch measurements of denitrification of the leachate water were performed during the last four years. The nitrogen load of each batch varied from 10 to 40 kg N (150 to 250 m$^3$ of drainage water in each batch). The nitrate nitrogen removal reached 3 to 4 kg N per day when there was excess carbon in the reactor (C/N ratio over 20:1). In most cases the denitrification rate was 0.5 to 1 kg N per day. The in situ batch denitrification cycles were measured over variable intervals ranging from 7 to 60 d.

Figure 4 shows a typical curve of nitrate and nitrite nitrogen removals, and particularly the intermediate formation of nitrites. Transient nitrite accumulation was observed in most of the in situ denitrification batches. Almost complete denitrification was achieved after 28 d (0.1 mg N-NO$_3$-l and 0.3 mg NO$_2$-l) but was pursued until zero nitrate and nitrite was reached by day 41. This represents more than 0.5 kg N-NO$_3$.
removal/day. The total Kjeldahl content of the water was controlled at three dates (beginning, middle and end of the denitrification cycle). The total nitrogen decreased from 126 mg N/l to 75 mg N/l. This decrease was of the same magnitude as the ammonium content and confirms that the nitrate and nitrite disappearance is owing to nitrogen gas (N$_2$) being produced and not to assimilatory reduction by bacterial or other aquatic organisms.

A second batch denitrification was started using half of the effluent from the previous batch study as bacterial seed. We added 106 m$^3$ of drainage water giving an average nitrate concentration of 31 mg N-NO$_3$/l in the reactor (total volume in the reactor 309 m$^3$). Complete denitrification was accomplished within 12 d.

3.4.2. Laboratory batch denitrification

During the batch study (Experiment 1), denitrification was complete after 4 d with the higher C/N ratio (8:2), and after 8 d with the C/N ratio of 2:5. The lower C/N ratio (0:5) was insufficient to maintain denitrification (Table 5). It was encouraging to note during our study that the pH of the two treatments with low C/N ratio (0 and 0:5) remained stable at values near neutral (6.5–7.2) whereas the two treatments with high C/N ratio (2:5 and 8:2) showed a similar increase in the pH, from 7:5 to 8:5. This observation confirms the denitrification process which is known to result in alkalinity. The BOD$_5$ removal during denitrification (63–99%) was always higher than the COD removal (56–73%) due to non-degradable organic matter applied with the slurry carbon source.

3.4.3. Laboratory semi-continuous denitrification

It was surprising to note that the treatment with the critical C/N ratio (0:5) that showed a failure during experiment 1 (batch) was able to achieve some additional denitrification during the semi-continuous treatment study shown in Table 5. This second experiment clearly demonstrates the feasibility of semi-continuous denitrification under our conditions. The yield of this consecutive denitrification was however dependent on the residual carbon available as shown in Fig. 5.

3.5. Nitrogen balance

After a one-year experiment, it was possible to establish a preliminary nitrogen balance from the slurry nitrogen applied through the soil/plant/water system. It showed that 44% of the N remained in the soil, 10% was taken up by the ryegrass, 5% leached and subsequently denitrified and 41% was lost from the soil/water/plant system, presumably due to ammonia volatilisation and/or soil denitrification. The nitrogen fluxes were further estimated (Fig. 6) and

<table>
<thead>
<tr>
<th>Initial C/N ratio</th>
<th>Nitrate nitrogen added (g)</th>
<th>Nitrate nitrogen removed (g)</th>
<th>% removal</th>
<th>Nitrate nitrogen added (g)</th>
<th>Nitrate nitrogen removed (g)</th>
<th>% removal</th>
</tr>
</thead>
<tbody>
<tr>
<td>0:5</td>
<td>5:4</td>
<td>1:45</td>
<td>27</td>
<td>4:48</td>
<td>0:49</td>
<td>11</td>
</tr>
<tr>
<td>2:5</td>
<td>5:6</td>
<td>5:59</td>
<td>100</td>
<td>4:71</td>
<td>1:49</td>
<td>32</td>
</tr>
<tr>
<td>8:2</td>
<td>5:2</td>
<td>5:17</td>
<td>99</td>
<td>4:66</td>
<td>4:66</td>
<td>100</td>
</tr>
</tbody>
</table>

Table 5

Influence of the initial C/N ratio on the efficiency of nitrate nitrogen removal during laboratory batch and subsequent semi-continuous runs

Fig. 5. Nitrate nitrogen removal versus nitrate nitrogen added depending on residual carbon available during a laboratory semi-continuous denitrification sequence (500 h); ■, Initial C/N ratio of 2:5 (low carbon availability); ▼, Initial C/N ratio of 8:2 (high carbon availability)
show particularly that the percentage nitrogen leached increased over the years and reached nearly 10% of the slurry nitrogen applied. The total nitrate nitrogen leached over years varied from 200 kg N/ha yr during drainage seasons 1991–92 and 1992–93, to 440 kg N/ha yr and 840 kg N/ha yr during drainage seasons 1993–94 and 1994–95 respectively. From 1991 to 1996, the nitrogen content of the top soil (0–20 cm depth) increased from 0.176% to 0.347%, which indicates an accumulation of 4450 kg N/ha (bulk density was established to be 1.3 g/cm³). The reserve of N accumulated for the three sampled layers (0–20, 20–40 and 40–60 cm) reaches 6080 kg N/ha which when compared with the 25 t/ha of total nitrogen applied during the five years of spreading gives about 25% immobilisation.

The ammonia volatilized following spreading was only estimated during the third year of slurry applications and was established to be 10% (Moal et al. 27). This process is however highly variable depending on weather and soil conditions and it was found that on some occasions, up to 50% of ammonia volatilized. The estimate given in Fig. 6 is thus a compromise between measured and literature data.

4. Discussion

A fully drained field such as Solepur with a total recovery of the leachate provided a unique opportunity to evaluate soil purification performances. The nutrient load in the Solepur field slurry purification facility is about twenty times that on normal agricultural land. However, the drainage water collected from the field was similarly clear to the drainage water taken from any agricultural land. This result illustrates the surprisingly high filtration and purification capacity of this silty sandy soil. Consequently, the physicochemical changes from soil composition shows a build-up of elements including nitrogen, carbon, potassium, phosphorus copper and zinc, mainly in the upper layers of soil (0–20 and 20–40 cm depth). The percentage recovery in the soil for these elements varied from 25% for total nitrogen to 43% for carbon, 41% for potassium, and reached 85–90% for phosphorus, copper and zinc. The question that arises from these results is for how long the soil will maintain its retention capacity as a physical and biological biobarrier. During a two-year study on the evaluation of the soil filter system for liquid dairy cattle wastes, Ritter and Eastburn19 established that the system was capable of reducing COD and nitrogen by 90% or more and phosphorus by 99%. The results from Solepur are consistent with this finding but the actual results were obtained over a longer experimental period of five years and with raw pig slurry as influent.

Nitrification is a two-step sequential aerobic oxidative pathway for the oxidation of ammonium (NH₄-N) to nitrate. This process occurs naturally in soils but at a rate compatible with soil N mineralisation (ammonification) usually in a range of 50 to 300 kg N/ha yr. Owing to the high ammonium supply to the Solepur soil from heavy repeated applications of slurry, it was necessary to ensure that this practice did not inhibit the nitrification potential of the soil. The results conclusively demonstrate that nitrifiers were still active in spite of the high nitrogen load applied. On average, over the four years, the soil received 312 g of ammonium-N/m² yr⁻¹. Couton et al. 28 described nitrogen removal from pig slurry based on a forced nitrification step using gravel columns. They obtained nitrification rates of 52 g N m⁻² d⁻¹ with a slurry input of 601 m⁻² d⁻¹. However in their study the residual ammonium-N in the percolates was high and the nitrification was not fully accomplished thus leading to nitrite accumulation. In our study the average slurry input was much lower (0.31 m⁻² d⁻¹) and nitrification was achieved with natural conditions. Since the autumn of 1992, after the first two years of intensive and repeated pig slurry applications, the nitrate concentration of the drainage water never fell below 300 mg/l, which indicated a continuous release and probably a continuous build up of nitrates in the soil due to mild winters in Brittany and Western France.

The drainage water denitrification by recycling carbon pig slurry is effective. In particular the influence of the C/N ratio proved to be the key factor for consideration. Several authors (McCarty et al. 29, Carley and Mavinic 30) have investigated the influence of the C/N ratio and carbon sources and observed similar
trends. In particular Carley and Mavinic\textsuperscript{30} established a range COD/N-NO\textsubscript{X} ratio of 5:9:1 to 9:0:1 to achieve complete denitrification (depending on the carbon source). In our study, the COD/N-NO\textsubscript{X} ratios for the four treatments evaluated were 0, 1:5:1, 6:9:1 and 20:7:1 respectively. So despite the differences in carbon source, which in the present study was pig slurry, it appears that a COD/N-NO\textsubscript{X} ratio of 6:1 is necessary to achieve a full denitrification sequence. Our results on semi-continuous denitrification also suggest that when excess carbon is used for a denitrification, it is possible to achieve a subsequent denitrification run by directly adding the nitrate rich leachate to the reactor, while a fraction of the total volume is left in the denitrification reactor as inoculum for the next batch without additional supply of pig slurry. The utilisation of this cheaper and available by-product to support denitrification makes it necessary to irrigate the denitrified effluent to a final soil filtration to remove the residual carbon load.

The indicative nitrogen balance also showed that an important part of the nitrogen budget amounting to 30–40% was unaccounted and was assumed to be lost by soil denitrification. It is probable that owing to the supply of carbon with the repeated slurry applications and the anaerobic zones created, the soil enhanced its denitrifying activity. The direct measurement of soil denitrification has been carried out recently (Chadwick, 1996; private communication of unpublished results) and tends to confirm that this process becomes important particularly during autumn and winter months when the soil is at field moisture capacity. It was also found that significant losses of N\textsubscript{2}O are emitted into the atmosphere. Other workers have investigated the manipulation of indigenous biological soil systems dynamics in order to reduce nitrate leaching from agricultural soils\textsuperscript{31} by adding a readily available carbon source, or by the addition of carbon directly to aquifers.\textsuperscript{32} These natural approaches have the potential to minimize agricultural pollution.

5. Conclusions

The Solepur unit is still, after five years of treatment, demonstrating the removal of organic matter and nitrogen from pig slurry with undiminished efficiency.

(1) With an annual nominal load of 1000 m\textsuperscript{3} ha\textsuperscript{-1} yr\textsuperscript{-1} of pig slurry, the first stage of the process, soil and biological filtration, led to the removal of 90% of nitrogen, 99.9% of phosphorus and reduced COD and BOD\textsubscript{5} to a similar extent.

(2) After five years of intensive and repeated pig slurry applications, and an annual nitrogen load in excess of 5000 kg N/ha, the Solepur soil treatment and nitrogen removal system maintains and even enhance its nitrification potential.

(3) The purification and biodegradation of raw pig slurry to a clear drainage water was satisfactorily demonstrated over 5 yr, which illustrates the strong filtration and purification capacity of the soil.

(4) The extent of nitrate rich leachate denitrification using small amounts of pig slurry as a carbon source was effective but require a final soil treatment stage for the denitrified effluent. A C/NO\textsubscript{3} -N ratio of 3:1 was found to be essential for complete denitrification of nitrate-rich leachate when raw pig slurry was used as an external carbon source.

(5) During both laboratory and field denitrification experiments, the phenomenon of transient accumulation of nitrite occurred, although it had been removed by the end of the denitrification process. This may lead to an increase in the production of nitrous oxide. Further studies of the denitrification gases are required.

(6) The approximate nitrogen balance showed that in situ soil denitrification during winter is probably an effective nitrogen removal pathway. However, operating conditions to avoid nitrous oxide emissions should be determined.

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