

## Anaerobic digestion of piggery waste. 1. The influence of detention time and manure concentration

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Accepted: 20 April 1977

**Key words:** Anaerobic digestion, piggery waste, odour control

### Summary

The influence of the manure concentration – in the range of 50 to 112.5 g total solids (TS)/litre – and the detention time – in the range of 40 to 10 days – upon the anaerobic digestion of piggery waste was investigated in laboratory experiments (1.5 - 6 litres) and in pilot plant experiments (240 litres). In spite of high ammonia nitrogen concentrations a stable digestion could be achieved and maintained at all manure concentrations and all detention times applied, except when 112.5 g TS/litre manure was added at a 10-day detention time (space load 8.1 kg volatile solids (VS) per m<sup>3</sup> per day).

Although a stable digestion could be maintained up to a space load of 5.4 kg VS m<sup>-3</sup> day<sup>-1</sup>, the maximum space load at which a satisfactory reduction of objectionable manure odour was attained appeared to be about 3.6 kg VS m<sup>-3</sup> day<sup>-1</sup>.

### Introduction

Intensive pig farming with its large concentrations of animals on a small space has led to a local excess of manure. The storage of the piggery waste, especially when urine and faeces are collected together, frequently causes odour nuisance, resulting in the need to treat the manure. Since conventional treatment of the excess animal waste, e.g. by means of aerobic stabilization, requires much energy, anaerobic digestion as a treatment method comes more and more into the picture.

In 1975 a research project was started in which the feasibility of piggery waste stabilization through anaerobic digestion was investigated. The primary objective of this project is to determine the reduction of objectional manure odour through anaerobic digestion. Additional goals included the determination of:

- process efficiency in recovering energy from piggery waste;
- process stability and process control, especially at changing process conditions;
- the nitrogen balance in connection with the use of the digested manure as fertilizer;

- total solids and volatile solids reduction;
- dewatering characteristics of the digested manure in relation to the raw manure.

Considering the practical application of methane fermentation for piggery waste stabilization, knowledge of the maximum achievable organic loading rate is necessary for optimum process design and process performance. Since the organic loading rate can be increased by increasing the total solids concentration of the feed manure as well as by decreasing the detention time, the effect of both parameters is described in this article.

## Material and methods

### *Methods*

*Analyses.* Gas analyses were performed by means of gas chromatography on a Fraktovap model M partitioner with a dual column arrangement of molecular sieve 30/60 mesh 5A and silica gel 30/60 mesh.

Chemical oxygen demand (COD) was estimated according to Standard Methods. Supernatant COD was determined after centrifugation at 14000 *g* for 10 minutes. Ammonia nitrogen was determined by steam distillation at pH 7.4 into boric acid indicator and titration with standard acid.

Volatile fatty acids (VFA) analyses were performed on a gas chromatograph, with a packed glass column (1 m × 0.4 cm i.d.) filled with Tween 80 on Chromosorb W-AW 80/100. Column temperature was 115 °C and the carrier gas, N<sub>2</sub>, was saturated with formic acid (Fohr, 1974).

Total solids concentration (TS) was determined by drying a 10 ml sample in a 105 °C stove overnight.<sup>1</sup> Volatile solids concentration (VS) was determined by glowing the dried samples to constant weight.<sup>1</sup>

Analysis of aromatic odorous components – phenol, p-cresol, ethylphenol, indole and skatole – was performed according to Spoelstra (1977).

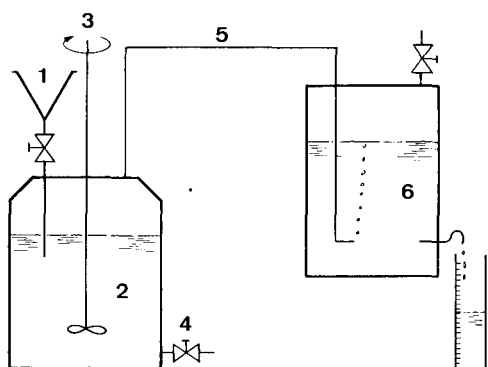
*Apparatus.* The experiments were carried out partly on a laboratory scale (1.5 - 6 litres) and partly on small pilot plant scale (240 litres). Although laboratory digesters with a volume of 1.5, 2, 5 and 6 litres were used, the experimental ar-

<sup>1</sup> Since some volatile matter may be lost (Miner & Smith, 1975) these analyses were checked by the following experiments.

– After entrapping the condensate vapours that are released during drying, the condensate was analysed to determine the ammonia nitrogen, VFA and COD concentrations. It turned out that all of the ammonia nitrogen and part of the VFA were found again in the condensate, whereas the COD value corresponded to the VFA concentration.

– To a digested manure sample containing no VFA a weighed amount of VFA, corresponding to the VFA concentration in raw manure, was added. The pH of the solution was comparable with the manure pH. Both solutions, with and without VFA, were analysed in triplicate to determine the TS concentration by drying at 105 °C overnight. The results indicated that 90 % of the VFA evaporated during the analysis.

Therefore all TS and VS analyses mentioned in this article are corrected as far as VFA (90 % lost) and ammonia nitrogen (all lost) are concerned.



1. inlet pipe
2. digestion vessel
3. stirring motor
4. outlet pipe
5. gas outflow pipe
6. brine displacement system

Fig. 1. Laboratory experimental digester system.

rangement was almost identical in all experiments (see Fig. 1). Gas production was determined by means of a brine displacement system (Fig. 1) using brine with a pH of 1 in the experiments with 1.5-litre and 2-litre digesters, and by means of a wet gas meter in the experiments with 5- and 6-litre digesters. The digestion units were placed in a temperature-controlled room of  $30 \pm 2^\circ\text{C}$ . Discontinuous agitation of the digester contents was accomplished by mechanical stirring for 15 s per 5 minutes at 100 rev/min.

The pilot plant consisted of two identical isolated digestion units having a volume of 240 litres each. The digester system is shown in Fig. 2. Fluid mixing was achieved by continuous gas recirculation with a gas flow of c. 3 litres/min. Furthermore the digester contents were mixed by recirculating the digester contents with a sludge pump (capacity c.  $5 \text{ m}^3/\text{h}$ ) for 15 minutes once a day. The daily feed was added during sludge recirculation. To prevent damage to the sludge pump the manure feed was sieved through a 0.5-cm sieve to remove hairs, straw, etc.

The digester heating system consisted of a hot water tank (temperature of water  $50^\circ\text{C}$ ), a water circulation pump and stainless steel heat exchange piping inside the digester. The digester temperature ( $32^\circ\text{C}$ ) was controlled by means of a thermo-couple and a thermostatic control, connected with the water circulation pump. The gas production was determined by a wet gas meter.

### Materials

The piggery waste used in the experiments consisted of urine and faeces and contained no or minor amounts of bedding or litter. The manure obtained from an experimental piggery farm, was supplied at a rather constant TS concentration of

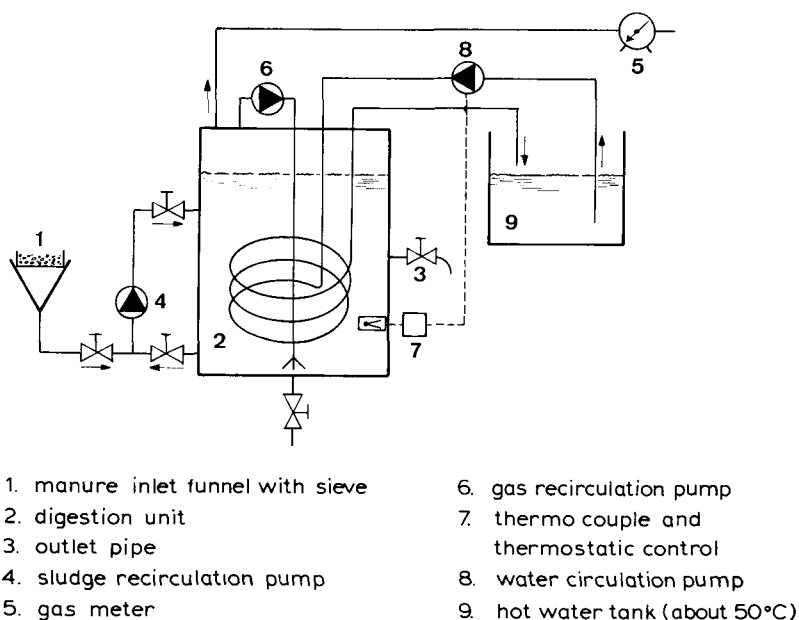


Fig. 2. Pilot plant system.

c. 130 g/litre and was diluted to the desired level by adding tap water. Table 1 summarizes the chemical composition of the manure after dilution to 75 g TS/litre. This concentration was used in most experiments except in some additional laboratory experiments where TS concentrations of 50 and 112.5 g/litre were applied.

The concentration of the aromatic odorous components in raw, diluted manure is shown in Table 2.

### Experimental procedure

The manure concentrations and the detention times applied in the experiments are given in Table 3. The first series of the laboratory experiments and the pilot plant experiments were seeded with digested sewage sludge, which was well adapted

Table 1. Chemical composition of piggery wast after dilution with tap water.

|         | TS<br>(g/litre) | VS<br>(g/litre) | NH <sub>4</sub> -N<br>(mg N/litre) | COD <sub>total</sub><br>(g O <sub>2</sub> /litre) | COD <sub>s</sub><br>(g O <sub>2</sub> /litre) | VFA<br>(meq/litre) |
|---------|-----------------|-----------------|------------------------------------|---|---|--------------------|
| Minimum | 73.5            | 52.1            | 1780                               | 64.5  | 17.5  | 107                |
| Maximum | 77.0            | 57.7            | 2635                               | 104.0   | 23.3  | 170                |
| Average | 74.5            | 54.0            | 2090                               | 80.3  | 19.4  | 134                |

TS = total solids; VS = volatile solids; COD = chemical oxygen demand; COD<sub>s</sub> = COD of the supernatant; VFA = volatile fatty acids.

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Table 2. Concentration of aromatic odorous components (in mg/litre) in 75 g TS /litre manure.

|         | Phenol | p-cresol | Ethylphenol | Indole | Skatole |
|---------|--------|----------|-------------|--------|---------|
| Minimum | 12.6   | 118.2    | 5.7         | 0.6    | 33.4    |
| Maximum | 55.4   | 195.6    | 14.4        | 3.7    | 48.7    |
| Average | 25.2   | 149.0    | 11.2        | 2.2    | 39.6    |

to piggery waste as it was fed with 75 g TS/litre piggery waste at a 40-day detention time for more than 6 months. In the second series of laboratory experiments seed sludge from the pilot plant experiment D<sub>2</sub> was used after a stable digestion at a 20-day detention time had been accomplished in this digester.

All digesters were fed daily except during the weekends, when only one feed was supplied, although of a double amount. The feeding procedure was as follows. After the determination of the gas production a prescribed volume (determined by the detention time) of the mixed digester contents was removed. Then the manure was added according to the organic and hydraulic load. Before and during the feeding procedure the digester contents were mixed. Because of this procedure the digesters can be considered to be completely mixed fermentors.

The analyses were carried out according to the time schedule shown in Table 4. The duration time of all experiments was at least three times the turn-over time in order to ensure a steady state and to avoid the influence of changing manure composition.

## Results

The results of the laboratory experiments – together with the experimental conditions applied – are summarized in Table 5 (manure concentration 50 g TS/litre, Table 6 (manure concentration 75 g TS/litre) and Table 7 (manure concentration 112.5 g TS/litre). The data in these tables concern the average values of the ana-

Table 3. Manure concentration and detention time of the experiments.

| Detention time<br>(days) | Manure concentration (g TS/litre) |                                     |                   |
|--------------------------|-----------------------------------|-------------------------------------|-------------------|
|                          | 50                                | 75                                  | 112.5             |
|                          |                                   | laboratory                          | pilot plant       |
| 40                       |                                   |                                     | D <sub>1</sub>    |
| 20                       |                                   | B <sub>1</sub> *, B <sub>2</sub> ** | D <sub>2</sub>    |
| 16                       |                                   | B <sub>3</sub> *                    | C <sub>1</sub> ** |
| 15                       | A <sub>1</sub> *                  |                                     | D <sub>3</sub>    |
| 12.5                     |                                   | B <sub>4</sub> *                    | C <sub>2</sub> ** |
| 12                       |                                   |                                     | D <sub>4</sub>    |
| 10                       | A <sub>2</sub> *                  | B <sub>5</sub> **                   | C <sub>3</sub> ** |

\* First series of experiments.

\*\* Second series of experiments.

Table 4. Analytical performance.

|                            | Daily | 3 times a week | Weekly | Fortnightly | Incidentally |
|----------------------------|-------|----------------|--------|-------------|--------------|
| Gas production             | ×     |                |        |             |              |
| Gas composition            |       |                | ×      |             |              |
| pH                         |       | ×              |        |             |              |
| COD <sub>total</sub>       |       |                |        |             | ×            |
| COD <sub>supernatant</sub> |       |                |        | ×           |              |
| VFA                        |       | ×              |        |             |              |
| Ammonia nitrogen           |       |                |        | ×           |              |
| Total solids               |       |                | ×      |             |              |
| Volatile solids            |       |                | ×      |             |              |
| Aromatic components        |       |                |        |             | ×            |

lytical results obtained during the last 4 weeks of the experiments, when digestion processes had reached a steady state. The values of the COD reduction mentioned in Tables 5 to 8 have been calculated from the amount of COD removed via methane gas and the initial manure COD (Table 1). The methane content of the digester gas, produced in Experiments A<sub>1</sub>, A<sub>2</sub>, B<sub>3</sub> and B<sub>4</sub>, is high in comparison

Table 5. Summary data at steady state operation of laboratory digesters, fed with 50 g TS/litre manure.

|   | Exp. A <sub>1</sub> | Exp. A <sub>2</sub> |
|---|---------------------|---------------------|
| Working digester volume (litres)  | 1.5                 | 1.5                 |
| Duration experiment (days)  | 92                  | 60                  |
| Detention time (days)   | 15                  | 10                  |
| Organic load: kg COD m <sup>-3</sup> day <sup>-1</sup>                        | 3.5                 | 5.3                 |
| kg VS m <sup>-3</sup> day <sup>-1</sup>                                       | 2.4                 | 3.6                 |
| Gas production (litres gas l <sub>r</sub> <sup>-1</sup> /day <sup>-1</sup> )* | 0.650               | 0.713               |
| Gas composition (% CH <sub>4</sub> )  | 84.9                | 82.8                |
| COD removal (g COD/litre manure)**  | 20.37               | 14.47               |
| Total COD reduction (%)   | 44.4                | 31.5                |
| <i>Digester effluent</i>  |                     |                     |
| pH  | 7.6                 | 7.45                |
| NH <sub>4</sub> -N (as mg N/litre)  | 1700                | 1832                |
| TS (g/litre)  | 33.2                | 37.4                |
| VS (g/litre)  | 20.7                | 24.4                |
| COD <sub>s</sub> (mg/litre)   | 6200                | 10000               |
| VFA (meq/litre)   | 12.9                | 38.6                |
| Acetic acid (meq/litre)   | 4.5                 | 4.1                 |
| Propionic acid (meq/litre)  | 4.1                 | 33.1                |
| TS reduction (%)  | 33.9                | 35.5                |
| VS reduction (%)  | 42.2                | 31.8                |
| COD <sub>s</sub> reduction (%)  | 63.1                | 40.0                |
| VFA reduction (%)   | 88.6                | 61.6                |

\* Gas production in litres of gas per litre working digester volume (l<sub>r</sub>).

\*\* Calculated from gas production and gas composition.

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Table 6. Summary data at steady state operation of laboratory experiments, fed with 75 g TS/litre manure.

|   | Exp. B <sub>1</sub> | Exp. B <sub>2</sub> | Exp. B <sub>3</sub> | Exp. B <sub>4</sub> | Exp. B <sub>5</sub> |
|---|---------------------|---------------------|---------------------|---------------------|---------------------|
| Working digester volume (litres)  | 6                   | 5                   | 2                   | 2                   | 6                   |
| Duration experiments (days)   | 102                 | 109                 | 92                  | 94                  | 93                  |
| Detention time (days)   | 20                  | 20                  | 16                  | 12.5                | 10                  |
| Organic load: kg COD m <sup>-3</sup> day <sup>-1</sup>                      | 4.0                 | 4.0                 | 5.0                 | 6.4                 | 8.0                 |
| kg VS m <sup>-3</sup> day <sup>-1</sup>                                     | 2.7                 | 2.7                 | 3.4                 | 4.3                 | 5.4                 |
| Gas production (litres gas l <sub>r</sub> <sup>-1</sup> day <sup>1</sup> )* | 0.897               | 0.810               | 0.930               | 1.032               | 1.300               |
| Gas composition (% CH <sub>4</sub> )  | 73.8                | 75.6                | 83.4                | 83.0                | 77.1                |
| COD removal (g COD/litre manure)**  | 32.57               | 30.13               | 30.53               | 26.34               | 24.66               |
| Total COD reduction (%)   | 44.6                | 41.7                | 41.7                | 36.4                | 34.2                |
| <i>Digester effluent</i>  |                     |                     |                     |                     |                     |
| pH  | 7.7                 | 7.8                 | 7.9                 | 7.8                 | 7.7                 |
| NH <sub>4</sub> -N (as mg N/litre)  | 2510                | 2740                | 2615                | 2580                | 2635                |
| TS (g/litre)  | 52.3                | 50.9                | 53.1                | 55.5                | 58.0                |
| VS (g/litre)  | 34.1                | 30.0                | 34.9                | 35.1                | 37.0                |
| COD <sub>s</sub> (mg/litre)   | 6750                | 5700                | 8500                | 9150                | 8600                |
| VFA (meq/litre)   | 3.1                 | 5.5                 | 10.7                | 17.6                | 23.4                |
| Acetic acid (meq/litre)   | 2.9                 | 5.5                 | 5.3                 | 10.0                | 11.0                |
| Propionic acid (meq/litre)  | 0                   | 0                   | 5.2                 | 7.3                 | 11.5                |
| TS reduction (%)  | 28.0                | 32.1                | 29.5                | 26.3                | 25.8                |
| VS reduction (%)  | 37.4                | 44.4                | 34.4                | 34.0                | 31.5                |
| COD <sub>s</sub> reduction (%)  | 75.0                | 75.5                | 66.0                | 63.4                | 63.0                |
| VFA reduction (%)   | 97.7                | 95.9                | 92.0                | 86.9                | 82.6                |

\* and \*\* See notes to Table 5.

with the gas produced in the other experiments. This difference has to be attributed to the use of a brine displacement system to collect the digester gas in the former experiments. In all laboratory digesters mixing was necessary to prevent the formation of a floating layer. Nevertheless, in some digesters a tough floating layer was formed, which could be disrupted by continuous stirring for 10 minutes. Scum formation appeared to occur only if the digestion process was not in balance. In none of the experimental units retarded digestion has been observed except in Experiment C<sub>3</sub>, where the feed consisted of concentrated manure (112.5 g TS/litre) and a detention time of 10 days was applied.

The conditions and results of the pilot plant experiments are summarized in Table 8. One digester was run at various detention times, viz 20, 15 and 12 days, whereas the other unit acting as a reference digester was run at a 40-day detention time.

In both digesters continuous gas recirculation was applied. In this way a uniform digester temperature could be achieved whereas the formation of a floating layer could be prevented. No scum formation was observed except during start-up periods and after digestion disturbance. Due to a failure in the temperature control system during experiment D<sub>2</sub>, the digester temperature was 50 °C for a period of

c. 24 hours. This resulted in a severe unbalance of the digestion process as was indicated by the sudden decrease of gas production, the low methane content of the digester gas and the strong increase of the VFA concentration.

To accomplish process recovery no feed was supplied for 3 days, followed by a period in which the quantity of the feed was increased in steps: for a period of 2 days 6 litres of manure, then for a period of 3 days 9 litres of manure, and subsequently with a daily quantity of 12 litres according to the original detention time of 20 days. The course of the process recovery is shown in Fig. 3. Although gas production and gas composition soon reached the original level the VFA concentration increased strongly, especially that of propionic acid. However, after 5 weeks the propionic acid concentration decreased rapidly, which indicated that the recovery of the digestion process had been completed.

As the objectionable manure odour has to be attributed to the presence of aromatic components, such as phenol, p-cresol, ethylphenol, indole and skatole, as well as VFA (Schaefer et al., 1974), the concentration of these components has been determined in raw manure as well as in the digested manure at the end of some experiments. The results of these analyses are shown in Fig. 4. When smelling the digester effluents it appeared that digested manure from the pilot plant experi-

Table 7. Summary data at steady state operation of laboratory experiments, fed with 112.5 g TS/litre manure.

|   | Exp. C <sub>1</sub> | Exp. C <sub>2</sub> | Exp. C <sub>3</sub> |
|---|---------------------|---------------------|---------------------|
| Working digester volume (litres)                                | 5                   | 5                   | 5                   |
| Duration experiment (days)                                      | 109                 | 109                 | 109                 |
| Detention time (days)   | 20                  | 15                  | 10                  |
| Organic load: kg COD m <sup>-3</sup> day <sup>-1</sup>          | 6.0                 | 8.0                 | 12.0                |
| kg VS m <sup>-3</sup> day <sup>-1</sup>                         | 4.1                 | 5.4                 | 8.1                 |
| Gas production (litres gas l <sup>-1</sup> day <sup>-1</sup> )* | 1.120               | 1.448               | 0.534               |
| Gas composition (% CH <sub>4</sub> )                            | 74.7                | 74.4                | 67.6                |
| COD removal (g COD/litre manure)**                              | 41.16               | 39.75               | 8.88                |
| Total COD reduction (%)   | 31.7                | 30.5                | 6.8                 |
| <i>Digester effluent</i>  |                     |                     |                     |
| pH  | 7.9                 | 7.9                 | 7.55                |
| NH <sub>4</sub> -N (as mg N/litre)                              | 3950                | 3910                | 3975                |
| TS (g/litre)  | 83.1                | 85.3                | 104.5               |
| VS (g/litre)  | 50.7                | 52.7                | 71.1                |
| COD <sub>s</sub> (mg/litre)                                     | 12000               | 13000               | 30600               |
| VFA (meq/litre)   | 18.9                | 20.1                | 201.3               |
| Acetic acid (meq/litre)   | 18.1                | 18.0                | 111.0               |
| Propionic acid (meq/litre)                                      | 0.8                 | 1.7                 | 63.5                |
| TS reduction (%)  | 27.1                | 25.2                | 8.3                 |
| VS reduction (%)  | 36.9                | 34.4                | 11.5                |
| COD <sub>s</sub> reduction (%)                                  | 66.4                | 63.6                | 14.3                |
| VFA reduction (%)   | 90.6                | 90.0                | 0.2                 |

\* and \*\* See notes to Table 5.



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Table 8. Summary data at steady state operation of pilot plant experiments, fed with 75 g TS/litre manure.

|   | Exp. D <sub>1</sub> | Exp. D <sub>2</sub> | Exp. D <sub>3</sub> | Exp. D <sub>4</sub> |
|---|---------------------|---------------------|---------------------|---------------------|
| Working digester volume (litres)                                | 240                 | 240                 | 240                 | 240                 |
| Duration experiment (days)                                      | 200                 | 100                 | 92                  | 40                  |
| Detention time (days)   | 40                  | 20                  | 15                  | 12                  |
| Organic load: kg COD m <sup>-3</sup> day <sup>-1</sup>          | 2.0                 | 4.0                 | 5.3                 | 6.7                 |
| kg VS m <sup>-3</sup> day <sup>-1</sup>                         | 1.4                 | 2.7                 | 3.6                 | 4.5                 |
| Gas production (litres gas l <sup>-1</sup> day <sup>-1</sup> )* | 0.420               | 0.850               | 1.030               | 1.180               |
| Gas composition (% CH <sub>4</sub> )                            | 73.9                | 75.2                | 76.4                | 76.3                |
| COD removal (g COD/litre manure)**                              | 30.54               | 31.45               | 29.04               | 26.58               |
| Total COD reduction (%)   | 49.0                | 44.3                | 43.9                | 40.3                |
| <i>Digester effluent</i>  |                     |                     |                     |                     |
| pH  | —                   | —                   | —                   | —                   |
| NH <sub>4</sub> -N (as mg N/litre)                              | 2965                | 2820                | 2675                | 2315                |
| TS (g/litre)  | 49.2                | 45.0                | 49.0                | 51.7                |
| VS (g/litre)  | 28.8                | 27.6                | 31.3                | 31.8                |
| COD <sub>s</sub> (mg/litre)                                     | 6600                | 6350                | 6550                | 6100                |
| VFA (meq/litre)   | 4.2                 | 4.2                 | 3.5                 | 4.7                 |
| Acetic acid (meq/litre)   | 4.2                 | 4.0                 | 3.5                 | 3.9                 |
| Propionic acid (meq/litre)                                      | 0                   | 0.2                 | 0                   | 0.8                 |
| TS reduction (%)  | 34.4                | 40.0                | 30.8                | 31.1                |
| VS reduction (%)  | 44.1                | 48.9                | 39.2                | 38.3                |
| COD <sub>s</sub> reduction (%)                                  | 61.2                | 74.1                | 62.9                | 65.5                |
| VFA reduction (%)   | 96.9                | 96.9                | 97.4                | 96.5                |

\* and \*\* See notes to Table 5.

ments did not have any of the original objectionable odours. However, digested manure from the laboratory units was less satisfactory in this respect.

### Discussion

Piggery waste, consisting mainly of carbohydrates, lipids and proteins, turned out to be a good substrate for anaerobic digestion. Successful digestion could be obtained at space loads from 1.4 to 5.4 kg VS m<sup>-3</sup> day<sup>-1</sup>. A space load of 8.1 kg VS m<sup>-3</sup> day<sup>-1</sup> as applied in experiment C<sub>3</sub>, however, resulted in a severe accumulation of VFA and therefore in retarded methane production. The values of the obtained organic load are in accordance with results reported by Loehr (1969), indicating that animal wastes could be successfully digested at loading rates in the range of 1.6 to 6.4 kg VS m<sup>-3</sup> day<sup>-1</sup>.

### Process efficiency

The COD reduction obtained in the pilot plants experiments varied from 40.3 % at a 12-day detention time to 49 % at a 40-day detention time, which is consider-

ably lower than the 54.6 % reported by Gramms et al. (1971). This also holds for the reduction of volatile solids. In our pilot plant experiments a VS reduction of 38.3 to 48.9 % was observed, whereas Gramms et al. (1971) reported a reduction value of 60.9 %. These differences may be attributed to the fact that the process conditions in the experiments of Gramms et al., such as the ammonia nitrogen concentration (190 - 570 mg N/litre) and the pH (6.76 - 7.27), differed from the conditions in our experiments.

In the laboratory experiments the VS reduction varied from 31.5 to 44.4 % at a balanced digestion. Furthermore, the results shown in Tables 5 to 8 indicate that the TS and VS reductions tend to decrease at decreasing detention times. However, due to sampling difficulties and possible erratic analytical methods (see analyses) the VS reduction is only a coarse parameter for describing animal waste digestion efficiency.

In stable digestion the gas production varied from 160 to 225 litres of gas per kg TS added, whereas the methane content of the digester gas was about 75 % when gas production was measured by means of a wet gas meter. The methane production as related to the total solids fed to the digesters has been plotted versus the detention time in Fig. 5. For comparison the methane production in Fig. 5 has

gas production (l gas/l r-day)

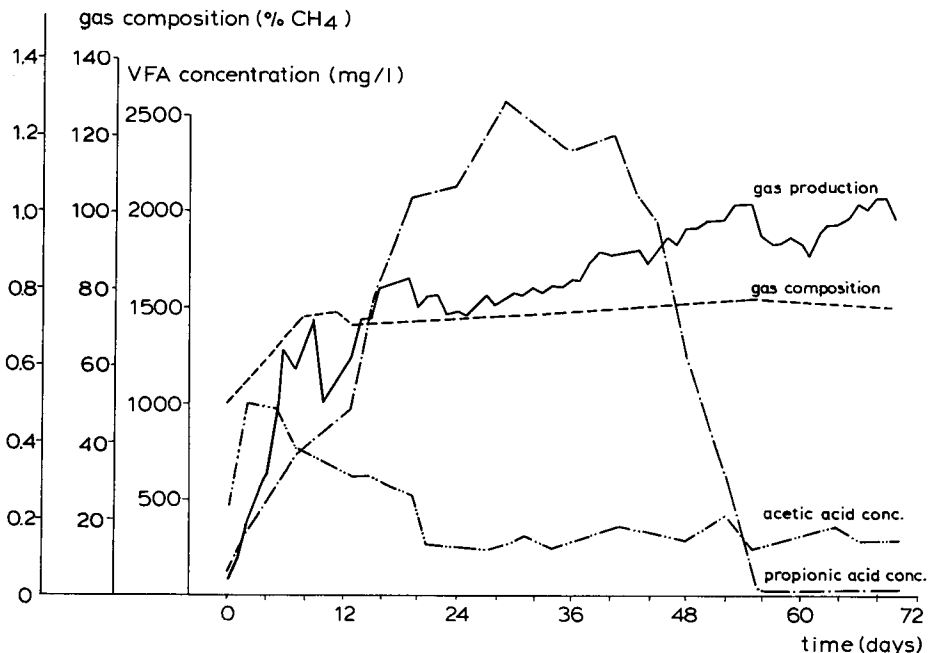


Fig. 3. Course of some process parameters after process disturbance due to a rise in temperature to 50 °C. For feeding rate see text.

relative concentration (%)

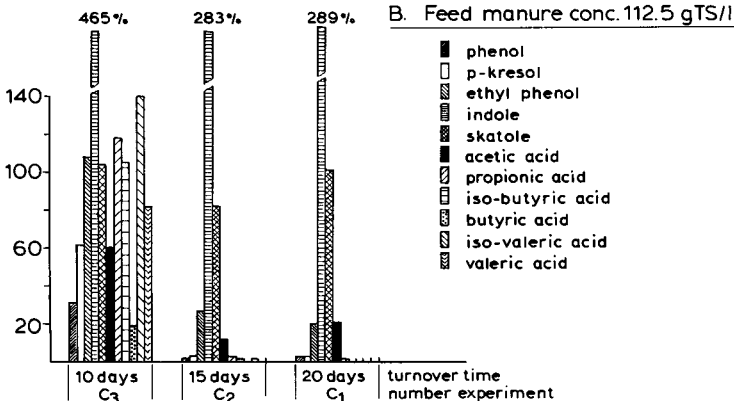
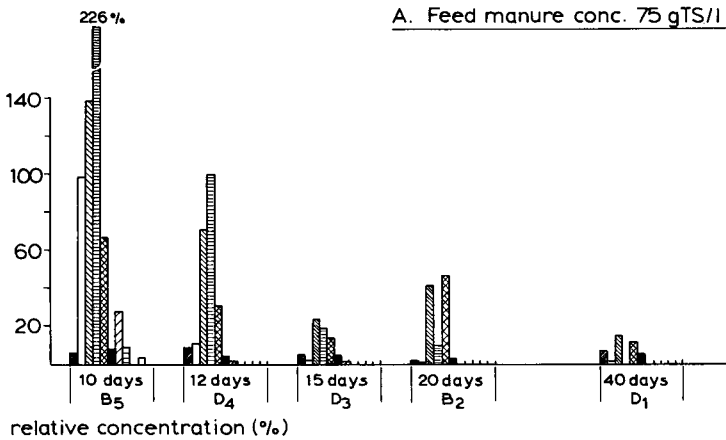


Fig. 4. Concentration of odorous components present in digested piggery waste. The concentration has been expressed in relation to the concentration in raw manure (see Table 2).

been expressed in terms of litres of methane produced per kg TS added. From Fig. 5 it appears that, at detention times longer than 15 days there is only a slight increase of the methane production (and consequently of the degree of stabilization of the manure) when manure concentrations of 75 and 112.5 g TS/litre are applied. As can be seen from Fig. 5, methane production decreases sharply at detention times shorter than 15 days in the experiments with 50 and 112.5 g TS/litre manure. A far less sharp decrease in methane production was observed in the experiments with 75 g TS/litre manure. Apparently a better process condition for a balanced digestion is obtained at a TS concentration of approximately 75 g TS/litre.

The effect, upon methane production, of the increase of organic load, either by increasing the TS concentration of the manure feed or by decreasing the detention time, is shown in Fig. 6. From Fig. 6 it appears that there is only a slight decrease

of methane production when the total solids concentration of the feed is increased at a 15-day detention time. This slight decrease in methane production, however, may also be attributed to the increased organic load. On the other hand Fig. 6 clearly shows that methane production is strongly affected by detention times in the range of 15 to 10 days.

The methane production was 400 - 600 litres of methane per kg VS removed, the average value being 540. This corresponds to a COD of 1.33 kg COD/kg VS removed. Apparently mainly volatile matter of a COD/VS ratio higher than 1, such as lipids, fatty acids and proteins, is converted.

#### *Ammonia nitrogen concentration ( $\text{NH}_4\text{-N}$ )*

An increase in the TS concentration of the feed implies an increase in the  $\text{NH}_4\text{-N}$  concentration in the digester fluid. Although  $\text{NH}_4\text{-N}$  concentrations over 3000 mg/litre were thought to be toxic for methane fermentation (McCarthy, 1964; Schmid & Ripper, 1969; Patel & Patel, 1971), especially in combination with a high pH level, the results of our experiments show that a balanced methane fermentation

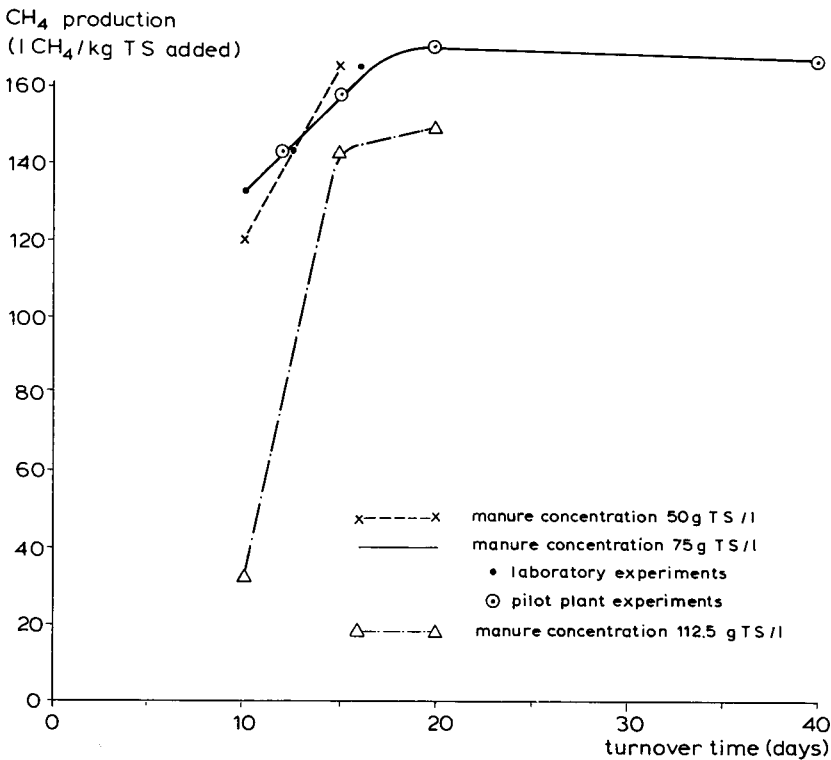


Fig. 5. Methane production vs. detection time at different manure feed concentrations.

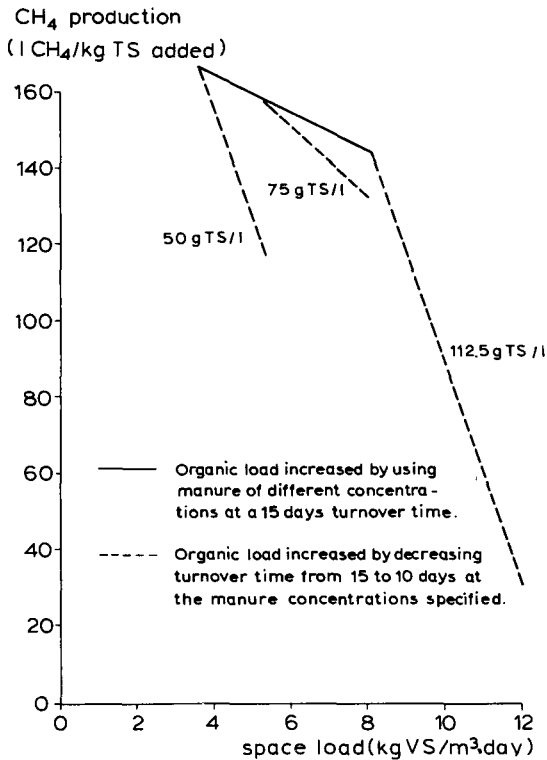


Fig. 6. Influence of the increase of organic load upon methane production.

takes place even at an  $\text{NH}_4\text{-N}$  concentration of 4000 mg/litre and a pH of 7.9 (Table 7). Perhaps even more interesting is the fact that the methane production rate is hardly affected by the  $\text{NH}_4\text{-N}$  concentration in the range of 1650 to 4000 mg/litre. At a 15-day detention time we observed a linear decrease in the methane production from 166 litres  $\text{CH}_4$ /kg TS added at an  $\text{NH}_4\text{-N}$  concentration of 1650 mg/litre to 144 litres  $\text{CH}_4$ /kg TS added at an  $\text{NH}_4\text{-N}$  concentration of 4000 mg/litre. Interpreting these results one has to consider that, at a constant detention time, an increase in the  $\text{NH}_4\text{-N}$  concentration is accompanied with an increase in the organic space load because of the more concentrated feed which has to be used. However, the influence of a high  $\text{NH}_4\text{-N}$  concentration upon the metabolism rate of the methanogenic bacteria does not become clear from these results, since methane formation is not the process rate-limiting step in these experiments, as will be discussed below. Successful digestion of piggery waste at  $\text{NH}_4\text{-N}$  concentrations as high as 3450 mg/litre has also been described by Kroeker et al. (1975). Melbinger & Donnellon (1971) reported that  $\text{NH}_4\text{-N}$  concentrations of 2700 mg/litre have existed in acclimated high rate digesters without affecting the gas production or the volatile matter destruction. According to Melbinger & Donnellon  $\text{NH}_4\text{-N}$  does not become toxic or inhibitive at concentrations over 1700-1800 mg/

litre, unless the rate of its formation increases more rapidly than the acclimation of methanogenic organisms. From the available data the impression is gathered that a bacterial population can adapt itself to a high  $\text{NH}_4\text{-N}$  concentration.

### *Reduction of odorous components*

The main odorous components present in piggery manure – VFA, phenol, p-cresol, ethylphenol, indole and skatole (Schaefer et al., 1974) – are formed through anaerobic microbiological activity. It is peculiar that these components also disappear under anaerobic conditions, as can be seen from Fig. 4.

The results of our experiments (Fig. 4) indicate that (1) elimination increases at increasing detention times; (2) the VFA are eliminated faster than the aromatic components; (3) a better elimination, especially of indole and skatole, is obtained at a low TS concentration in the digester fluid.

In the experiments with 50 g TS/litre manure a decrease of the detention time from 15 to 10 days resulted in an increase in the VFA concentration, especially that of the propionic acid concentration (Table 5). The concentrations of phenol, p-cresol, ethylphenol, indole and skatole were not determined in these experiments.

When, in the pilot plant experiments, manure with 75 g TS/litre was used, the VFA concentration was 3.5 to 4.2 meq/litre at all detention times examined, viz 40, 20, 15 and 12 days (Table 8), whereas in the laboratory experiments the VFA concentration increased from 3.1 meq/litre at a 20-day detention time to 23.4 meq/litre at a 10-day detention time (Table 6). In these experiments, too, the increase of the VFA concentration was caused mainly by an increase in the propionic acid concentration. Of the aromatic components, phenol is almost completely eliminated within 10 days (see Fig. 4), whereas the reduction of p-cresol increased from 2 % at a 10-day detention time to 89 % at a 12-day detention time. An explanation for the remarkably better elimination rate at a 12-day detention time may be (1) the organisms responsible for the elimination of p-cresol have – under these conditions – a generation time between 10 and 12 days, or (2) at a 12-day detention time all easily degradable constituents of the manure feed have been reduced to a low concentration, which gives micro-organisms the opportunity of metabolizing less degradable substrates, such as some aromatic components. In the experiments with a 10-day detention time the indole concentration became as high as 266 % in comparison with the indole concentration in the raw manure, which indicates that its formation rate surpasses the elimination rate. When, however, the detention time is increased the indole elimination rate becomes higher than the formation rate and at a 40-day detention time indole has disappeared completely.

When the results of Experiments  $D_3$  and  $D_1$  are compared with the results of Experiment  $B_2$  (see Fig. 4) it again appears that in laboratory scale experiments the reduction of odorous components is lower than in the pilot plant experiments. Accordingly the quality of the pilot plant effluents was much higher than the quality of the laboratory digester effluents with respect to objectionable odour.

In the experiments with 112.5 g TS/litre manure the VFA concentration was c. 18 meq/litre at a detention time of both 20 and 15 days (Table 5). At a 10-day

detention time no VFA reduction occurred at all. The propionic acid concentration in the digestion liquid was even higher than in the manure feed. In spite of a very high VFA concentration of 200 meq/litre in this experiment the digester pH remained 7.55 due to the high buffer capacity of the solution. In all experiments with 112.5 g TS/litre manure no elimination of indole and skatole occurred, not even at a 20-day detention time. This poor result may be due to the fact that high space loads were applied, which leads to an acceptable elimination of only the easily degradable organic matter.

### *Process balance*

As volatile fatty acids can be considered to be intermediates in the conversion of complex organic matter into methane and carbon dioxide, the VFA concentration is a useful indicator of process balance (Kroeker et al., 1975; McCarthy, 1964). The VFA concentration in the pilot plant experiments – in these experiments almost exclusively consisting of acetic acid – was 3.5 - 4.2 meq/litre. In our laboratory experiments the VFA concentrations in stable digestion units were in the range of 3.1 to 38.6 meq/litre. At a 20-day detention time acetic acid was the only volatile fatty acid present, whereas at decreasing detention times the increase of the VFA concentration was mainly caused by an increase in the propionic acid concentration. With respect to the recovery of the digestion process after the process disturbance mentioned above (see Fig. 3) we found that much more time was involved in the recovery of the propionic acid degradation than in that of other VFA. Hobson et al (1974) also observed that at failing digestion propionic acid was the principal VFA. From all these observations it appears that propionic acid is a critical factor in anaerobic digestion of piggery waste and that the course of the propionic acid concentration is a more useful indicator of process balance than the total VFA concentration.

### *Process rate-limiting step*

From the point of view of process optimization it is important to get a clear insight into the separate biochemical steps of the digestion process, viz hydrolysis, acid formation and methane formation. For this purpose it is assumed that (1) in hydrolysis undissolved organic matter<sup>1</sup> is converted into dissolved fragments; (2) VFA, hydrogen and carbon dioxide are exclusively formed by acid-forming bacteria; (3) all methane produced originates from the end-products of acid formation. The degree of hydrolysis, acid formation and methane formation can now be calculated by the following equations:

$$\text{Hydrolysis (\%)} = 100 (G + S)/M.$$

$$\text{Acid formation (\%)} = 100 (G + V)/M.$$

$$\text{Methane formation (\%)} = 100 G/M.$$

<sup>1</sup> Arbitrarily defined as the material that is precipitated during centrifugation at 14000 g for 10 minutes.

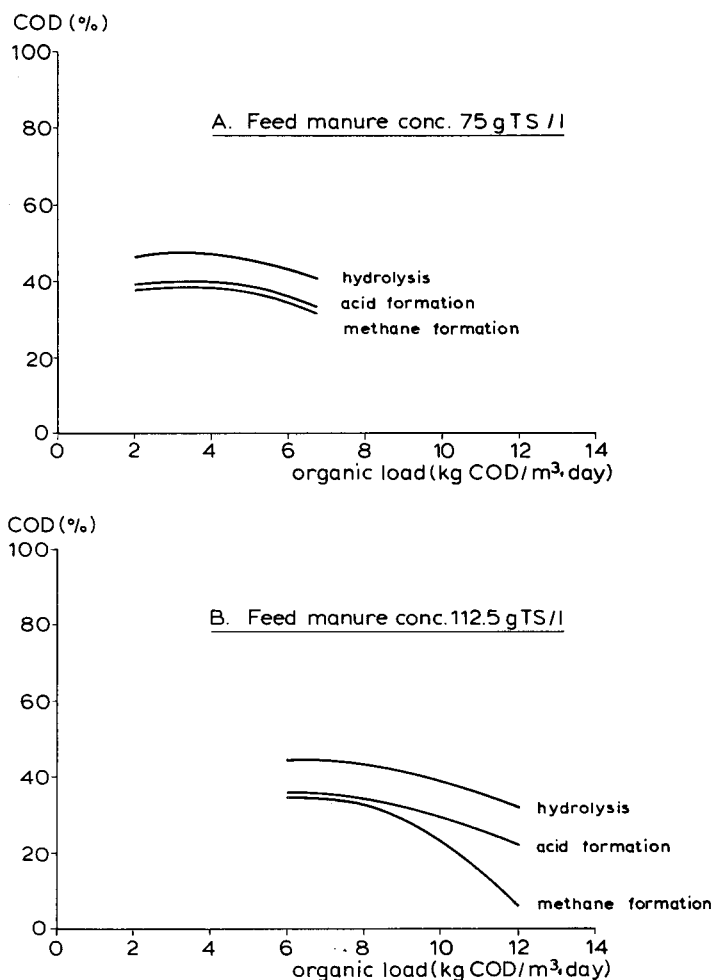


Fig. 7. Influence of the organic load upon hydrolysis, acid formation and methane formation.

in which:  $G$  = COD removed via methane gas ( $\text{g O}_2/\text{litre manure}$ )  
 $S$  = COD supernatant ( $\text{g O}_2/\text{litre}$ )  
 $M$  = total manure COD ( $\text{g O}_2/\text{litre manure}$ )  
 $V$  = COD, corresponding with the VFA concentration in the digester effluent ( $\text{g O}_2/\text{litre}$ ).

The results of these calculations have been plotted versus the organic load in Fig. 7a (pilot plant experiments) and Fig. 7b (laboratory experiments, using a manure concentration of 112.5 g TS/litre). The results of the pilot plant experiments indicate that at a decrease of the detention time in the range of 40 to 12 days the rate of the biochemical steps decrease to the same extent. These results lead



to the conclusion that the first step in the anaerobic conversion – viz hydrolysis – is the rate-limiting step in the overall process. Hobson et al. (1974) and Maki (1954) have already suggested that hydrolysis is the rate-limiting factor when organic matter is digested. According to Maki the cellulose hydrolysis in particular could be one of the rate-limiting steps in anaerobic digestion. In the experiments with 112.5 g TS/litre manure (Fig. 7b) hydrolysis is again the rate-limiting step when the detention time is decreased from 20 to 15 days. When the detention time is decreased from 15 days (space load  $5.4 \text{ kg VS m}^{-3} \text{ day}^{-1}$ ) to 10 days (space load  $8.1 \text{ kg VS m}^{-3} \text{ day}^{-1}$ ) the methane formation rate decreases far stronger than the hydrolysis and acid formation rates. This indicates that methane formation is the rate-limiting step. Acid formation was never found to be the rate-limiting step in the anaerobic fermentation of piggery waste.

### *Practical application*

Considering the practical application of anaerobic fermentation for the stabilization of piggery waste, the most important requirements are (1) a satisfactory reduction of odorous components, and (2) optimum process economics. Optimum process economics are obtained when a high space load is combined with a maximum gas production. From the results presented in this article it appears that a balanced digestion can be performed at a space load of c.  $5.4 \text{ kg VS m}^{-3} \text{ day}^{-1}$ , either by digesting 112.5 g TS/litre manure at a 15-day detention time resulting in a methane production of 144 litres  $\text{CH}_4/\text{kg TS}$  added or by digesting 75 g TS/litre manure at a 10-day detention time resulting in a methane production of 133 litres  $\text{CH}_4/\text{kg TS}$  added. However, at a space load of  $5.4 \text{ kg VS m}^{-3} \text{ day}^{-1}$  the reduction of odorous components is less satisfactory. In order to create a situation in which both requirements are met, the optimum space load must be c.  $3.6 \text{ kg VS m}^{-3} \text{ day}^{-1}$ , provided that this load is obtained by digesting 75 g TS/litre manure at a 15-day detention time. This will result in a methane production of 157 litres  $\text{CH}_4/\text{kg TS}$  added (see Table 6).

### **Conclusions**

1. Piggery waste, consisting of combined urine and faeces, is amenable to anaerobic digestion. Balanced digestions have been obtained at space loads in the range of 1.6 to  $5.4 \text{ kg VS m}^{-3} \text{ day}^{-1}$  and detention times of 10 days at the minimum. A retarded digestion occurred at a loading rate of  $8.1 \text{ kg VS m}^{-3} \text{ day}^{-1}$  and a 10-day detention time.
2. An almost optimum digestion for obtaining a satisfactory reduction of odorous components is achieved at a 75 g TS/litre concentration of the piggery waste at a 15-day detention time.
3. When the space load is increased, the methane production is more strongly affected by the detention time than by the TS concentration of the manure feed.
4. The propionic acid concentration is a useful indicator of process stability.
5. At decreasing detention times the methane production per kg VS added de-

creases, mainly because hydrolysis – which is the primary rate-limiting step – becomes incomplete. At a high organic load, viz 8.1 kg VS m<sup>-3</sup> day<sup>-1</sup>, the methane formation becomes the rate-limiting factor.

6. It appears that the anaerobic digestion of piggery waste is hardly affected by high ammonia nitrogen concentrations (as high as 4000 mg NH<sub>4</sub>-N/litre) in combination with a pH of 7.9.

7. Indole and skatole are the odorous components with the slowest elimination rate. The reduction of these components decreases (1) at decreasing detention times and (2) at increasing TS concentrations of the manure feed.

### Acknowledgments

The author wishes to thank G. Lettinga (Dept. of Water Purification, Agricultural University, Wageningen, the Netherlands) for his close co-operation and S. Spoelstra (Dept. of Microbiology, Agricultural University, Wageningen, the Netherlands) for the use of his method of analysing aromatic components. Part of the work was conducted at the Central Institute for Nutrition and Food Research (CIVO-TNO), Zeist, the Netherlands. J. de Waart provided the use of the facilities there and his co-operation is acknowledged.

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