



PERGAMON

www.elsevier.com/locate/watres

Wat. Res. Vol. 34, No. 2, pp. 673–677, 2000  
© 1999 Elsevier Science Ltd. All rights reserved  
Printed in Great Britain  
0043-1354/99/\$ - see front matter

PII: S0043-1354(99)00166-9

## RESEARCH NOTE

# THE INVERSE TURBULENT BED: A NOVEL BIOREACTOR FOR ANAEROBIC TREATMENT

PIERRE BUFFIÈRE\*, JEAN-PIERRE BERGEON and RENÉ MOLETTA<sup>M</sup>

INRA, Laboratoire de Biotechnologie de l'Environnement, Avenue des Etangs, 11100 Narbonne, France

(First received 1 April 1998; accepted in revised form 1 April 1999)

**Abstract**—An inverse turbulent bed was investigated for anaerobic digestion. In this reactor, a granular floating solid is expanded by an up-flow current of gas. The carrier particles (Extendspheres<sup>®</sup>) were chosen for their large specific surface area ( $20000\text{ m}^2\text{ m}^{-3}$ ) and their low energy requirements for fluidization (gas velocity of  $1.5\text{ mm s}^{-1}$ ,  $5.4\text{ m h}^{-1}$ ). It was tested with wine distillery wastewater and presented promising performances after 90 days of operation: 75–85% carbon removal with an organic loading rate of  $15\text{ kg}_{\text{COD}}\text{ m}^{-3}\text{ day}^{-1}$ . The amount of biomass (in terms of attached volatile solids) was close to  $0.2\text{ g g}^{-1}$  of solid at the end of the tests. Most particles were covered with a thin biofilm of uniform thickness, which was attributed to the friction effects due to turbulence. The performance observed during the start-up was similar or even higher than that of other previously tested fluidized bed technologies treating the same wastewater. © 1999 Elsevier Science Ltd. All rights reserved

**Key words**—inverse turbulent bed, anaerobic digestion, granular floating material, reactor start-up

### NOMENCLATURE

|       |   |
|-------|---|
| AVS   | attached volatile solids ( $\text{kg m}^{-3}$ or $\text{kg kg}_{\text{carrier}}^{-1}$ ) |
| COD   | chemical oxygen demand ( $\text{kg m}^{-3}$ )   |
| $d_s$ | clean particle diameter (m)   |
| $d_p$ | bioparticle diameter (m)  |
| $L_f$ | biofilm thickness (m)   |
| OLR   | organic loading rate ( $\text{kg}_{\text{COD}}\text{ m}^{-3}\text{ day}^{-1}$ )         |
| TOC   | total organic carbon ( $\text{kg m}^{-3}$ )   |
| VFA   | volatile fatty acids ( $\text{kg m}^{-3}$ )   |

### INTRODUCTION

Fluidized bed biofilm reactors are now commonly adapted to wastewater treatment. Literature is rich in successful reports on this technology. It is used either for aerobic treatment (Sutton, 1980; Shieh and Keenan, 1986; Nikolov and Karamanev, 1991; Rusten *et al.*, 1995), or anaerobic treatment such as denitrification (Hancher *et al.*, 1978; Green *et al.*, 1994) and anaerobic digestion (Bull *et al.*, 1983; Jeris, 1983; Heijnen *et al.*, 1989; Anderson *et al.*, 1990). In the field of anaerobic digestion, however, it has not been extensively used at full scale. This lack of industrial success may result from a combination of several negative points: a high level of

maintenance because of their complexity, the need for liquid recycling, or hydrodynamic problems (Buffière *et al.*, 1998; Schwarz *et al.*, 1998).

More recently, investigations were carried out on inverse fluidized beds (Shimodaira and Yushina, 1983; Nikolov and Karamanev, 1991; Chan Choi *et al.*, 1995; Garcia-Calderon *et al.*, 1998b). In this kind of reactors, floating particles are fluidized by a downflow current of liquid (see Fig. 1a). This technology presents interesting features: the downflow configuration enables overcoated particles to be recovered in the bottom of the bed. Moreover, the liquid and the biogas are flowing in opposite directions, which helps for bed expansion (Garcia-Calderon *et al.*, 1998a).

The expansion of a floating carrier is also possible under an upflow current of gas only (Fig. 1b). This phenomenon (called pseudo-fluidization) has already been identified by several teams (Fan *et al.*, 1982; Chern *et al.*, 1983; Legile *et al.*, 1988; Comte *et al.*, 1997). The gas bubbles generate downward liquid motions and apparent bed expansion. Theoretically, this kind of reactor may enable a stable control of the bed height, since the particles cannot settle below the gas injection zone because of their density. This work summarizes the first experimental steps of the development of an anaerobic inverse turbulent bed, through an application on wine distillery wastewater treatment.

\*Author to whom all correspondence should be addressed.  
Tel.: +33-4-6842-5168; fax: +33-4-6842-5160; e-mail: buffiere@ensam.inra.fr

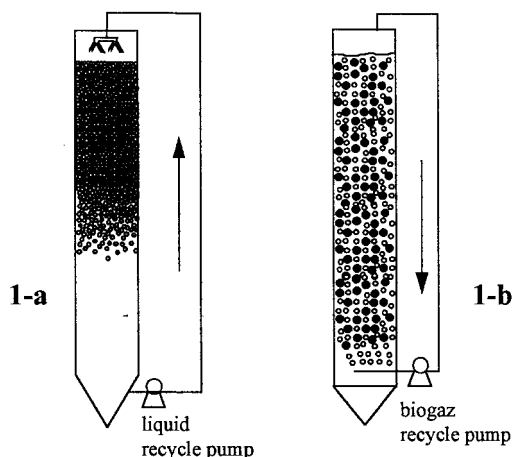


Fig. 1. The inverse fluidized bed (a) and the inverse turbulent bed (b).

## MATERIALS AND METHODS

### Experimental device

The experimental device used in this study is depicted in Fig. 2. The reactor consists in a tubular section of 0.08 m internal diameter and 0.8 m height with a conic bottom. The system is equipped with a water jacket keeping the liquid temperature at  $35 \pm 2^\circ\text{C}$ . The biogas is recycled by a pump at a constant flow rate of  $0.51 \text{ l min}^{-1}$  (gas superficial velocity of  $6 \text{ m h}^{-1}$ ,  $1.65 \text{ mm s}^{-1}$ ). The gas injection point is located 0.2 m above the bottom of the reactor and the gas is delivered through a perforated rubber tube. The gas production is measured by a gas flow meter. The reactor is monitored for pH and addition of NaOH is provided when the pH drops below 7. The substrate is an industrial wine distillery wastewater with a concentration of  $8\text{--}12 \text{ kg}_{\text{TOC}} \text{ m}^{-3}$  ( $20\text{--}30 \text{ kg}_{\text{COD}} \text{ m}^{-3}$ ).

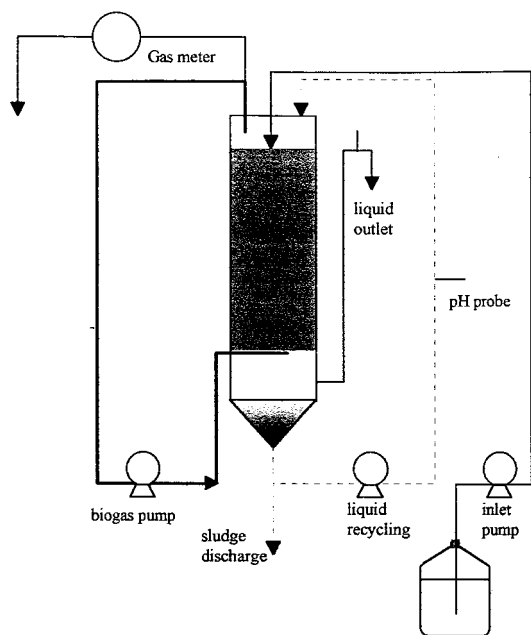


Fig. 2. Experimental anaerobic inverse turbulent bed bioreactor.

### Reactor inoculation and start-up

The reactor was filled with the solid carrier material (Extendsphere<sup>®</sup>,  $d_p = 175 \mu\text{m}$ ,  $\rho_s = 690 \text{ kg m}^{-3}$ ) up to 40% of its active volume. Extendsphere<sup>®</sup> (provided by PQ Hollowsphere) is a light mineral granular material mainly composed of silica. The shape of the particle is perfectly spherical, and the surface of the material present small crevices. It requires a low gas velocity for being expanded ( $1.5 \text{ mm s}^{-1}$ ). An increase of the gas velocity (that can be expected in full scale reactors with intense gas production) does not affect the fluidization, since the flow regime remains the same below  $10 \text{ mm s}^{-1}$ . The liquid inoculum was a mixing of anaerobic sludge and anaerobically treated distillery wastewater in a ratio of 1:2. The gas-recycling loop of the reactor was filled with nitrogen in order to ensure anaerobic conditions. During the first two weeks of acclimatization, a constant liquid recycling from the bottom to the top of the reactor was kept in order to provide a good contact between the (settling) biomass and the (floating) solid carrier. The liquid recycle ratio was about 2 (corresponding to a negligible liquid velocity of about  $40 \text{ mm day}^{-1}$ ) and was shut down when a consistent biogas production began.

### Measurements and analysis

Volatile fatty acids, total organic carbon and biogas composition were determined daily through off-line analysis. Volatile fatty acids (VFA) were measured with a flame ionization detector gas chromatograph Chrompack CP 9000. Total organic carbon was measured with a Dohrmann DC 80 carbon analyzer. Biogas content was analyzed with a catharometer gas chromatograph Shimadzu GC 8A. Total and volatile solids in the output and within the reactor were measured according to standard methods. Attached volatile solids were measured on washed samples of bioparticles. The average biofilm size on particles was estimated from diameter measurements. The measurement was performed with an optical microscope and a Leitz-Wetzlar graduated slide. The diameter was determined for approximately 50 particles for each sample. The measurements were done with clean and coated particles in order to estimate the biofilm size as the semi-difference between the average diameters,  $L_f = (d_p - d_s)/2$ .

## RESULTS AND DISCUSSION

### Reactor start-up

The 90 first days of operation of the reactor are summarized in Fig. 3. Fig. 3a is a plot of the VFA and alkalinity (expressed in  $\text{g}_{\text{CaCO}_3}/\text{l}$ ) concentrations at the outlet of the reactor. Fig. 3b represents the evolution of biogas and methane production (expressed in  $\text{l}/\text{l}_{\text{reactor}}/\text{d}$ ) and methane content of the gaseous phase. Fig. 3c is the plot of input organic load (expressed in  $\text{kg}_{\text{COD}} \text{ m}^{-3} \text{ day}^{-1}$ ) and carbon removal efficiency. The start-up period is divided into two parts. From day 1 to day 20, the influent wastewater was taken directly from the storage tank without any addition of nutrients, but very few microbial growth occurred and the input organic loading rate (OLR) had to be kept low ( $2 \text{ kg}_{\text{COD}} \text{ m}^{-3} \text{ day}^{-1}$ ). In the same time, the VFA concentration rose up to  $6 \text{ g/l}$  and the carbon removal failed down to 65%. From day 21 (arrow on the graph), a mixture of trace elements (Mg, Co, Cu, Li, ...) was added to the inlet together with a

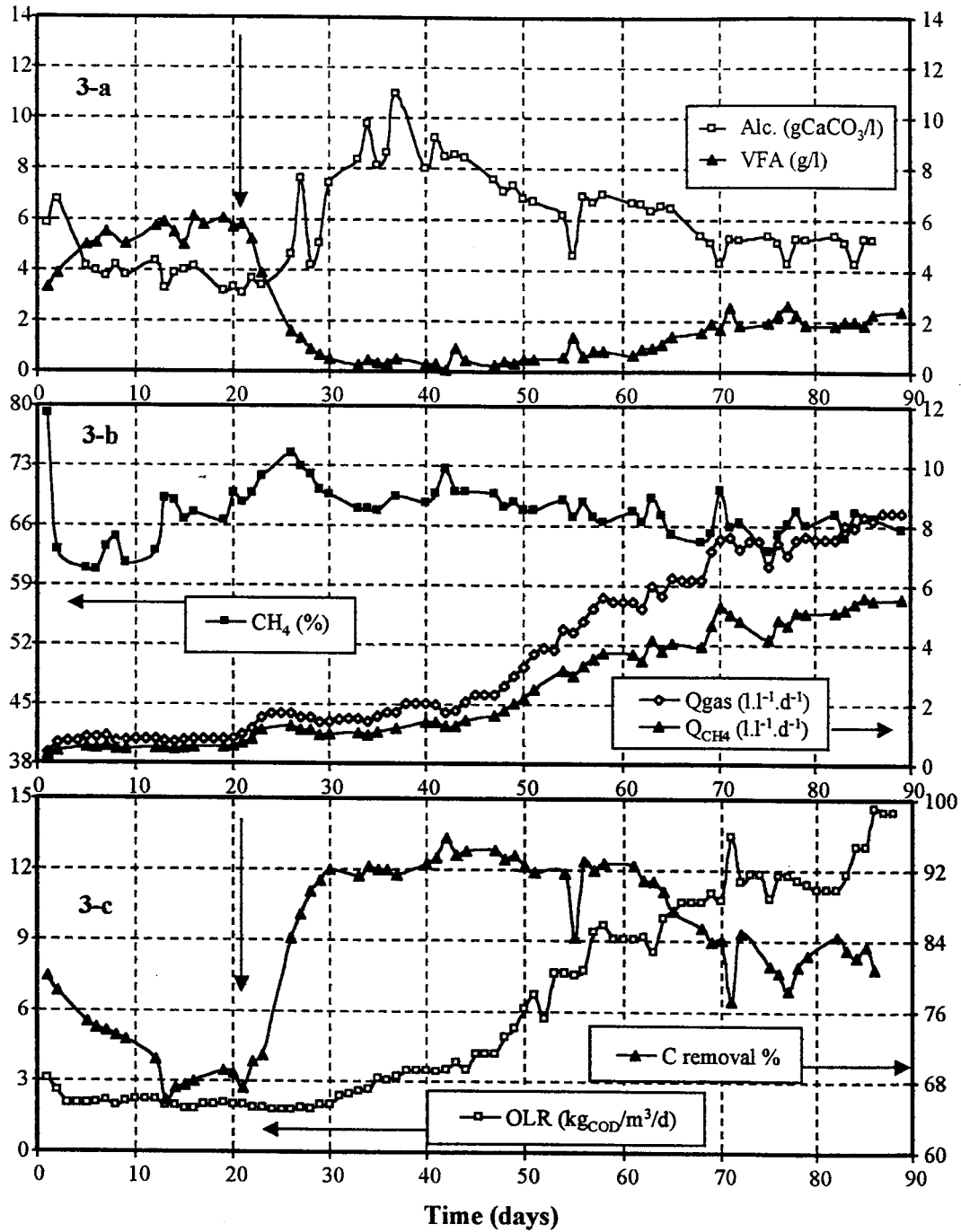


Fig. 3. Reactor startup.

nitrogen source. Within 4 days, the reactor had recovered an excellent carbon removal efficiency (over 90%). The VFA concentration dropped down to 0.2–0.3 g/l and the methane content in the gaseous phase increased. Further investigations carried out on batch tests for biodegradation of the effluent revealed that the problem came rather from trace elements than from nitrogen limitations. The OLR could then be increased from 2 to 15 kg<sub>COD</sub> m<sup>-3</sup> day<sup>-1</sup> over the following two months

of operation. When the OLR reached 10, the efficiency was partly affected: the carbon removal reached 76–85% and the VFA amount slightly rose up to 1.8–2.2 g/l. The CH<sub>4</sub> content in the gaseous phase was stabilized around 64–70%. The strategy was then to wait for the stabilization (or slight decrease) of the VFA concentration and to increase the input flow rate. From day 70 to day 86, the input loading rate could be increased from 10 to almost 15 without any noticeable change in the

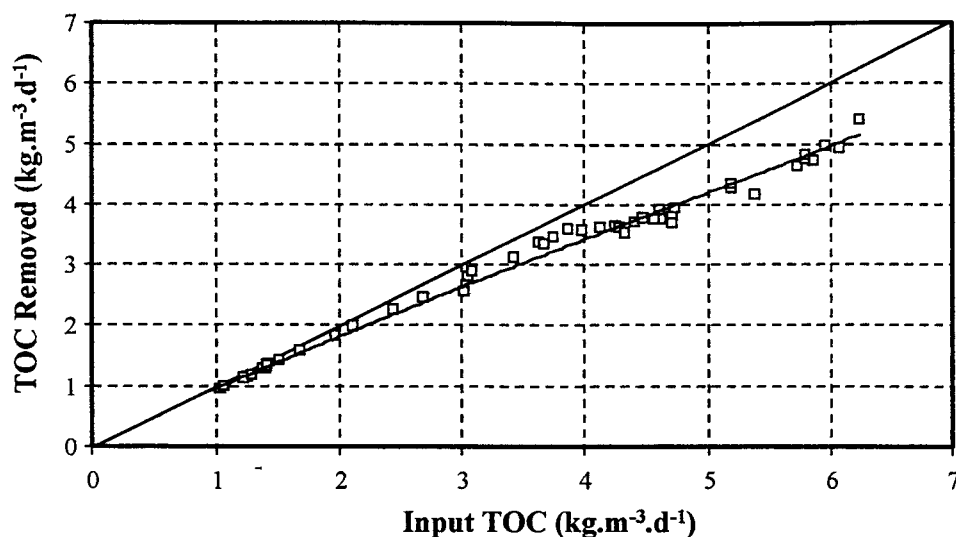


Fig. 4. Relationship between TOC loading and TOC removed.

measured control parameters. The biogas and methane production rates at the end of the experiment were 8.5 and 5.61 l<sub>reactor</sub><sup>-1</sup> day<sup>-1</sup> respectively.

#### *Biomass growth and carrier colonization*

Over the first 20 days, very few changes occurred in the reactor concerning the aspect of the carrier particles. The adjunction of trace elements and nitrogen source in the influent mixture quickly modified the aspect of the reactor. First of all, the color of the particles obviously turned from almost white to intense gray. Microscopic observations confirmed the presence of biomass on the carrier, either as local outgrowth colonies, either as uniform covering of the particles.

The evolution of the biofilm colonization versus time may be characterized as follows. On day 20, hardly 10% of the particles had a colony on its surface. On day 80, 50% approximately of the bioparticles were not fully covered.

The biomass coverage on the particles is very uniform: a large number of particles are covered regularly by a thin biofilm of constant thickness. This may be due to their perfectly spherical shape and to the friction effects enhanced by turbulence.

The amount of attached volatile solids (AVS) per gram of carrier could be measured once a reasonable biomass amount was established on the particles. On day 70, it was 0.177 g<sub>AVS</sub>/g<sub>solid</sub>, and 0.202 on day 80. This means that the reactor is capable of high biomass retention, and thus of higher performances, as the colonization phase was not finished on day 90. The average biofilm thickness at the end of the experiment was approximately 13 μm. In order to illustrate this, the amount of carbon removed versus the amount of carbon introduced into the reactor is plotted in Fig. 4 (expressed in TOC). It follows a quasi straight line with a slight inflexion

for organic loading closed to 4–5 kg<sub>TOC</sub> m<sup>-3</sup> day<sup>-1</sup>. This inflexion is not confirmed by the last points. This means that the limit of the system was not reached. If it was, the curve should level off at high carbon amounts.

#### *Discussion*

Over the 90 first days of operation, the first 20 days can be considered as “lost” for the process start-up, because of the nutrient deficiency in the wastewater used and the poor resulting biomass growth. The initial startup period could thus have been reduced to 60 days. Nevertheless, this would still remain long for a digester.

A comparison with previously studied reactors treating the same kind of effluents can be done. The initial start-up is similar to what we observed with a classical up-flow fluidized bed working with 385 μm pozzolana particles (Buffière *et al.*, 1995). In this experiment, the OLR was increased from 2 to 18 kg<sub>COD</sub> m<sup>-3</sup> day<sup>-1</sup> in 70 days, and the carbon removal varied between 92 and 75%. In an inverse fluidized bed with a downflow liquid fluidization using perlite as biomass carrier, the OLR was increased from 3 to 15 in 60 days, but the reactor was destabilized and the carbon removal was only 55% at the end of the experiment and the input load had to be decreased (Garcia-Calderon *et al.*, 1998a). The amount of attached biomass for the up-flow fluidized bed was 0.17 g<sub>AVS</sub> g<sub>carrier</sub><sup>-1</sup> at the end of the start-up period (90 days), 0.18 g g<sup>-1</sup> after 160 days with the inverse liquid fluidized bed, and 0.2 g g<sup>-1</sup> in the present case after 90 days (perlite particles having a density of 0.71 compared to 0.69 for Extendspheres<sup>®</sup>).

Further investigation could be done on this kind of reactors. For instance, the amount of solid to be used in the reactor has been fixed here at 40% of

the working volume. Other workers report that a correct fluidized state can be obtained for filling rates as high as 60–70%, all the more since the minimal fluidization velocity is lower for high solid amounts (Comte *et al.*, 1997). This question is crucial as far as the reactor has to be optimized. First, the influence of initial filling rate on biomass growth and attrition is not known. Second, increasing the solid amount in the reactor mechanically reduces the liquid residence time, and thus the contact between the liquid and the biomass; it also affects the value of the gas hold-up. The relationship between the hydrodynamic conditions, biomass retention, friction loss and overall carbon removal rate still needs investigations.

### CONCLUSION

This work intended to test a new kind of anaerobic digester with attached biomass, the inverse turbulent bed. In this reactor, the originality arises from the use of a floating solid carrier for microbial adhesion, and a fluidization ensured by an up-flow current of gas. This reactor has proved good abilities for wine distillery wastewater treatment.

The inverse turbulent bed presented several advantages compared to classical high rate digesters. First, bed height control results automatically from the location of the gas injection device. Second, the bottom of the reactor can be used as a settler for recovering the sludge or the overcoated particles. Third, a gas injection in a reactor is easier than a liquid recycling: very few clogging problems are met.

The application to the treatment of a wine distillery wastewater presented satisfactory results compared to other reactors (up-flow and down-flow classical fluidized beds). The input organic loading rate could be increased from 2 to 15 kg<sub>COD</sub> m<sup>-3</sup> day<sup>-1</sup> within less than 90 days, despite a 20 days delay due to a nutrient deficiency problem. The carbon removal was kept around 75–85%, and the hydraulic retention time could be reduced down to 1 day.

The main perspectives of this study concern the relationship between the hydrodynamic conditions in the reactor, the biomass growth onto the solid carrier and the influence on overall reactor performance. Further hydrodynamics studies are needed to complete this approach.

### REFERENCES

- Anderson G. K., Ozturk I. and Saw C. B. (1990) Pilot scale experiences on anaerobic fluidized bed treatment of brewery wastes. *Water Sci. Tech.* **22**, 202–209.
- Buffière P., Fonade C. and Moletta R. (1995) Continuous operation of a fluidized bed bioreactor for anaerobic digestion: residence time influence on degradation kinetics. *Biotech. Letters* **17**(8), 833–838.
- Buffière P., Fonade C. and Moletta R. (1998) Mixing and phase hold-up variations due to gas production in anaerobic fluidized bed digesters: influence on reactor performance. *Biotech. Bioeng.* **60**(1), 36–43.
- Bull M. A., Sterritt R. M. and Lester J. N. (1983) Response of the anaerobic fluidized bed reactor to transient changes in process parameters. *Water Res.* **17**(11), 1563–1568.
- Chan Choi Y., Seog Kim D., Joo Park K. and Koo Song S. (1995) Wastewater treatment in a pilot scale inverse fluidized bed biofilm reactor. *Biotech. Tech.* **9**(1), 35–40.
- Chern S.-H., Muroyama K. and Fan L.-S. (1983) Hydrodynamics of constrained inverse fluidization and semifluidization in gas–liquid–solid system. *Chem. Eng. Sci.* **38**(8), 1167–1174.
- Comte M.-P., Bastoul D., Hebrard G., Roustan M. and Lazarova V. (1997) Hydrodynamics of a three-phase fluidized bed — the inverse turbulent bed. *Chem. Eng. Sci.* **52**(21/22), 3971–3977.
- Fan L.-S., Muroyama K. and Chern S. H. (1982) Hydrodynamic characteristics of inverse fluidisation in liquid–solid and gas–liquid–solid systems. *Chem. Eng. J.* **24**, 143–150.
- Garcia-Calderon D., Buffière P., Moletta R. and Elmaleh S. (1998a) Anaerobic digestion of wine distillery wastewater in downflow fluidized bed. *Water Res.* **32**(12), 3593–3600.
- Garcia-Calderon D., Buffière P., Moletta R. and Elmaleh S. (1998b) Influence of biomass accumulation on bed expansion characteristics of a down flow anaerobic fluidized bed reactor. *Biotech. Bioeng.* **57**(2), 136–144.
- Green M., Shnitzer M., Tarre S., Bogdan B., Shelef G. and Sorden C. J. (1994) Fluidized bed reactor operation for groundwater denitrification. *Water Sci. Tech.* **29**(10/11), 509–515.
- Hancher C., Taylor P. and Napier J. (1978) Operation of a fluidized bed bioreactor for denitrification. *Biotech. Bioengng. Symp. Ser.* **8**, 361–378.
- Heijnen J. J., Mulder A., Enger W. and Hoeks F. (1989) Review on the application of anaerobic fluidized bed reactors in wastewater treatment. *Chem. Eng. J.* **41**, B37–B50.
- Jeris J. S. (1983) Industrial wastewater treatment using anaerobic fluidized bed reactors. *Water Sci. Tech.* **15**, 169–176.
- Legile P., Menard G., Laurent C., Thomas D. and Bernis A. (1988) Contribution à l'étude hydrodynamique d'un lit fluidisé triphasique fonctionnant à contre-courant. *Entropie* **143–144**, 23–31.
- Nikolov L. and Karamanev D. (1991) The inverse fluidization — a new approach to biofilm reactor design, to aerobic wastewater treatment. *Studies Environ. Sci.* **42**, 177–182.
- Rusten B., Hem L. and Ødegaard H. (1995) Nitrification of municipal wastewater in moving-bed biofilm reactors. *Water Environ. Res.* **67**(1), 75–86.
- Schwarz A., Hammer J., Mösche M., Jordening H.-J., Buchholz K. and Reuss M. (1998) Interactions between reaction engineering and fluid dynamics for an industrial scale fluidized bed reactor for anaerobic wastewater treatment. In *Proc. Waste Decision '98, Narbonne, France*.
- Shieh W. K. and Keenan J. D. (1986) Fluidized bed biofilm reactor for wastewater treatment. In *Advances in Biochemical Engineering*, ed. A. Fietcher, pp. 131–169. Springer Verlag.
- Shimodaira C. and Yushina Y. (1983) Biological wastewater treatment with downflow fluidized bed reactor. In *Proc. of the 3rd Pacific Chemical Engineering Congress, Seoul, Korea*, pp. 237–242.
- Sutton P. M. (1980) Application of the Oxitron system fluidized bed process to industrial wastewater treatment. In *Advances in Biotechnology*, ed. M. Moo-Young, pp. 607–613. Pergamon Press.