



# Brazilian Journal of Chemical Engineering Print version ISSN 0104-6632

Braz. J. Chem. Eng. vol. 14 no. 1 São Paulo Mar. 1997

http://dx.doi.org/10.1590/S0104-66321997000100001

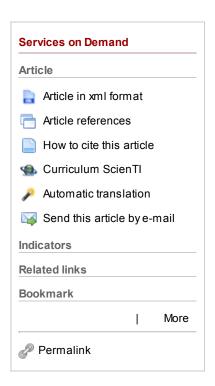
RATIONAL BASIS FOR DESIGNING HORIZONTAL-FLOW ANAEROBIC IMMOBILIZED SLUDGE (HAIS) REACTOR FOR WASTEWATER TREATMENT

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(Received: July 4, 1996; Accepted: October 18, 1996)



Abstract - The conception and development on a rational basis of a new configuration of anaerobic fixed-bed bioreactor for wastewater treatment, the horizontal-flow anaerobic immobilized sludge (HAIS) reactor, is presented. Such a reactor containing immobilized sludge in polyurethane foam matrices was first assayed for treating paper industry wastewater. A very short start-up period was observed and the reactor achieved stable operation by the eighth day. Afterwards, fundamental aspects of the process were investigated in order to obtain a rational basis for HAIS reactor design. A sequence of experiments was carried out for evaluating the cell wash-out from polyurethane foam matrices, the liquid-phase mass transfer coefficient and the intrinsic kinetic parameters, besides the hydrodynamic flow pattern of the reactor. The knowledge of such fundamental phenomena is useful for improving the reactors design and operation. Besides, these fundamental studies are essential to provide parameters for simulation and optimization of processes that make use of immobilized biomass. Keywords: HAIS reactor, mass transfer, kinetic, hydrodynamic, design.

# **INTRODUCTION**

Bioreactors containing immobilized anaerobic bacteria have been developed aiming at the decrease of the total unit volume since cell immobilization permits the improvement of process stability and performance, even at low hydraulic detention times (HDT). In fact, immobilized cell reactors are known to permit continuous operation without biomass washout and also to increase the time available for the catalytic function of cells in a reaction or in a series of reactions (Fan, 1989). The development of most of the anaerobic reactor configurations containing immobilized sludge is essentially based on empirical criteria. The predominance of empirical over rational criteria arises as a consequence of the variety and complexity of interactive processes occurring in such heterogeneous units. As a consequence, the study of fundamental phenomena can be very difficult. Moreover, the results obtained are often inadequate for design, scale-up or operational control. Even so, the knowledge of some fundamental aspects of the process such as intraparticle and liquid-phase mass transfer, intrinsic kinetic parameters and reactor hydrodynamics would be useful for improving reactor design and operation. Besides,

fundamental studies are essential for providing parameters for simulation and optimization of processes that make use of immobilized biomass.

A new configuration for anaerobic fixed-bed bioreactors, the horizontal-flow anaerobic immobilized sludge (HAIS) reactor, proposed by Zaiat et al. (1994), was developed on a rational basis. Studies on a bench-scale unit provided parameters and relationships for scale-up, simulation and optimization of the reactor.

#### CONCEPTION OF THE BENCH-SCALE HAIS REACTOR

The horizontal-flow anaerobic immobilized sludge (HAIS) bench-scale bioreactor is comprised of a 1 m long glass tube of 0.05 m diameter provided with a perforated tube of 0.9 cm diameter installed at its upper part for gas separation and collection. The reactors total volume is 2 liters. The HAIS scheme is shown in <u>Figure 1</u>.

The HAIS reactor was filled with polyurethane foam cubic matrices (side of 3-5 mm)

containing anaerobic immobilized sludge and it is meant to be a predominantly plug-flow regime reactor. The axial mixing due to the formation and vertical rise of gas across the horizontal liquid flow, as well as the tubular form of the reactor, is expected to promote a plug-flow like regime behaving as N-mixed reactors in series. Moreover, the gas tube collector along the reactor permits the minimization of dead volume for gas separation.

#### **HYDRODYNAMIC CHARACTERIZATION**

Cabral (1995) reported on stimulus-response experiments on a bench-scale HAIS reactor, aiming to verify its hydrodynamic characteristics. An additional method was developed to neglect the effect of the tracer-effective diffusion in the porous media on residence time distribution (RTD) curves.

The results from these experiments, shown in Figure 2, lead to the conclusion that the N- CSTR in series and the low and high dispersion theoretical models were well adjusted to the experimental data. It was demonstrated that the HAIS reactor can be simulated by approximately 30 continuous stirred tank reactors (CSTR) in series. This behavior suggests that the HAIS reactor is a "plug-flow" reactor for designing purposes. It was also found that the tracer effective diffusion in the particles' bed interferes strongly in the RTD curves and, consequently, in the parameters of the theoretical models. This may cause misinterpretation of the experimental data, resulting in design and scale-up errors.



Figure 1: Scheme of the bench-scale horizontal-flow anaerobic immobilized sludge (HAIS) reactor.

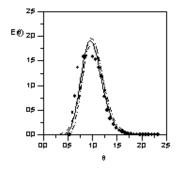


Figure 2: RTD curve obtained from hydrodynamic experiment in HAIS reactor operating with mean detention time of 2.4 hr and gas production of 1136 ml/day. Theoretical models adjusted to experimental data (◆ ): N-CSTR in series (— ), low dispersion (---) and high dispersion (- -).

# **PERFORMANCE TESTS**

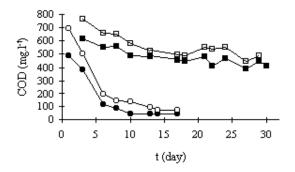
A HAIS reactor containing immobilized sludge in polyurethane foam matrices was first assayed for treating paper industry wastewater (Foresti et al., 1995). The reactor was operated at ambient temperature during 26 days at a constant hydraulic detention time (HDT) of 9.2 hours, based on the bulk liquid volume and mean organic loading rate (OLR) of 2.0 Kg COD/m³.d. Considering the net liquid volume, the OLR applied was 5.0 Kg COD/m³.d. A very short start-up period was observed and the reactor achieved stable operation by the eighth day. During the

15/03/13 Brazilian Journal of Chemical Engineering - Rational Basis for Designing Horizontal-Flow Anaerobic Immobilized Sludge (HAIS) Reactor for Waste... experiment, the average chemical oxygen demand (COD) removal efficiency was 82% at an HDT of 9.2 hours at the average temperature of 23° C.

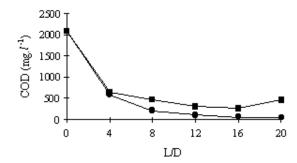
Other performance tests were carried out with the lab-scale HAIS reactor treating synthetic substrate to observe temporal and spatial variations of the performance parameters (Zaiat et al., 1996a). The knowledge of the behavior of such parameters along the reactor's length provides an important tool for the optimization of the length to diameter ratio (L/D), which can be considered the key design parameter of HAIS reactors, while their temporal variations provide information on the start-up period process evolution.

The bench-scale HAIS reactor was filled with polyurethane foam matrices containing immobilized anaerobic sludge taken from an up-flow anaerobic sludge blanket (UASB) reactor treating diluted piggery wastewater. A synthetic substrate with glucose as the main carbon source (COD of 2090 mg/l) was used for feeding the HAIS reactor in two experiments with bed porosities ( $\epsilon$ ) of 0.4 and 0.24, at an HDT of 8.0 and 4.8 hours, respectively. The temperature was maintained at 30° C, and COD, volatile fatty acids (VFA), total alkalinity (TA) and pH were monitored in the effluent stream (L/D of 20) and intermediate ports along the reactors length (L/D of 4, 8, 12 and 16). Data on COD temporal and spatial variations are shown in Figures 3 and 4, respectively. A short start-up period duration (6 days) in both the experiments confirmed that a HAIS reactor can provide favorable environmental conditions for rapid biomass acclimatization, growth and retention. COD removal efficiencies of 98% were attained in the first experiment ( $\varepsilon = 0.4$ ), in which effluent VFA concentration fell to 15 mg/l. In the second experiment ( $\epsilon = 0.24$ ), the maximum COD removal efficiency was 80% and the effluent VFA leveled off at 350 mg/l. Samplings along the reactors length were performed after the tenth day. The constant values of filtered COD and VFA concentrations along with the experimental time for all the L/D sample ports confirmed that the steady-state regimen had already been established.  $E_{COD}$  attained 90% for an L/D of 8 (HDT = 3.2 hr) and 97% for an L/D of 16 (HDT = 6.4 hr) for  $\varepsilon$  = 0,4. Therefore, the maximum E<sub>COD</sub> was practically obtained for an HDT of 6.4 hours. The effluent COD concentrations (L/D = 20) were higher than those obtained in some intermediate ports along the reactors length during the operation with  $\varepsilon = 0.24$ . This was an important indication of channeling in the reactor, permitting the conclusion that instantaneous samples from intermediate ports were not representative of the overall section, due to axial gradients of flow and concentration. Therefore, channeling might have occurred in the upper part of the reactor (gas-liquid interface) due to low bed porosity.

The behavior of the monitoring performance parameters along the HAIS reactors length observed in both the experiments is in accordance with the "ideal plug-flow" regimen foreseen by hydrodynamic studies. Such a well-defined hydrodynamic characteristic makes the HAIS reactor attractive for studies aiming to elucidate some still obscure aspects related to the dynamics of anaerobic mixed-cultures. Moreover, the low degree of longitudinal mixing in the reactor indicates the possibility of its use for some specific purpose. For example, wastewaters containing toxic compounds could be treated in a segregated part of the HAIS reactor filled with immobilized sludge enriched with microrganisms able to degrade such compounds. In that case, toxic compounds would not affect the overall biomass inside the reactor. Another advantage of the plug-flow pattern is that any intermediate product from bacterial metabolism that would be toxic or recalcitrant could be removed from the liquid phase by able or acclimatized microrganisms in the region of the reactor where such compounds would be predominantly produced. Therefore, HAIS reactors could be designed and operated for preventing microrganism activity inhibition by substrate or intermediate products. This hydrodynamic behavior would also permit the addition of aerated steps along the HAIS reactor for nutrient removal or for effluent polishment (Zaiat et al., 1996a).



**Figure 3:** Temporal variation of effluent filtered ( $\bullet$  , n ) and raw (o, ) COD in HAIS reactor operating with  $\epsilon$  of 0.4 and 0.24, respectively.



**Figure 4:** Spatial variation of filtered COD in HAIS reactor operating with  $\varepsilon$  of 0.4 ( $\bullet$ ) and 0.24 (n).

#### **CELL WASH-OUT FROM POLYURETHANE FOAM MATRICES**

Experiments were carried out to verify the effect of liquid superficial velocities ( $v_s$ ) on the wash-out of anaerobic biomass immobilized in polyurethane foam matrices (Zaiat et al., 1996b). Cell wash-out assays were carried out using a 10 ml tube segment of 1.5 cm diameter to permit the attainment of high liquid superficial velocities. This tube segment was filled with immobilized anaerobic sludge matrices and water was pumped from a reservoir to the tube through a peristaltic pump, at 8 different flow-rates which increased gradually. After each increase, the flow rate was maintained for 15 minutes, the full  $v_s$  range being from 0.30 cm/s to 2.21 cm/s.

The results showed a significantly high TSS loss for the initial  $v_s$  (0.30 cm/s) applied, indicating the presence of a weakly attached solid fraction in the matrices. Part of these solids probably might have become loosened during the tube segment filling operation. The TSS wash-out rate continuously decreased between the  $v_s$  of 0.83 cm/s and the  $v_s$  of 1.97 cm/s. However the total TSS loss was considerable (23.2%).

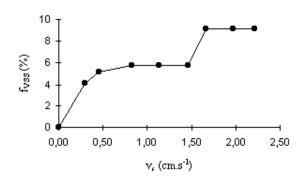


Figure 5: Washed-out VSS accumulated fraction ( $f_{VSS}$ ) as a function of liquid superficial velocity ( $v_s$ ).

On the contrary, the VSS losses were not significant until the  $v_s$  value of 0.83 cm/s (<u>Figure 5</u>). Moreover, from 0.83 cm/s to 1.66 cm/s, the losses were not detected, possibly because they were lower than the accuracy of the VSS determination method. However, a high VSS loss occurred at a  $v_s$  of 1.66 cm/s and, again, no VSS losses could be determined beyond this  $v_s$  value. It is seen that liquid superficial velocities of approximately 1.5 cm/s are critical for cell wash-out, but even so there was no considerable loss of VSS (9%). These results indicate that the microrganisms were firmly attached to the polyurethane foam matrices.

The difference between the observed TSS and VSS losses should be better investigated in the future, since it indicates that the wash-out action was selective. It is possible that the anaerobic sludge inert fraction tends to be loose inside porous matrices, since the cellular material is known to develop some type of linkage with inert surfaces. It is also possible that some bacteria species do not attach firmly, and remain dispersed inside the matrices. However, it has to be emphasized that the values of  $v_s$  applied to the tube segment were more than 20 times higher than those applied to the HAIS reactor in previous research (Foresti et al., 1995).

### MASS TRANSFER AND KINETICS

Methodologies for estimating the intrinsic kinetic parameters for immobilized anaerobic sludge used for wastewater

treatment and the external mass transfer coefficient in the bed of polyurethane foam were developed by Zaiat et al. (1996c, 1996d) and Vieira et al. (1996). The apparatus for the experiments on external mass transfer and kinetics of substrate utilization consisted of a 10 ml differential reactor of 1.5 cm length filled with immobilized sludge matrices. A volume of 300 ml of a glucose-based substrate prepared according to Del Nery (1987) with a COD of 228 mg/l was pumped from an agitated vessel to the reactor. Before entering the reactor, the feeding solution was heated to  $30^{\circ}$  C and then returned to the storage vessel maintained at  $5^{\circ}$  C to prevent the occurrence of biochemical reactions within it. The substrate was sterilized to maintain its initial characteristics in each experiment.

The applied flow rates permitted the liquid superficial velocity ( $v_s$ ) to be varied between 0.007 and 0.075 cm/s. Each experiment lasted 10 hours and effluent sampling was done every 2 hours for soluble COD analysis. Each assay allowed the obtainment of a respective COD profile, according to the value of the applied  $v_s$ . The overall reaction rate (r) was obtained as a function of the COD for each applied  $v_s$ :

$$r = -\frac{d(Sb)}{dt}$$
 (1)

where  $S_b$  is the COD concentration in the bulk liquid and t is the time.

### **Liquid-Phase Mass Transfer**

Considering the liquid-phase mass transfer as the limiting of the overall process rate, the volumetric coefficient of mass transfer  $(k_sa)$  can be estimated by:

$$r = k_s a. (S_b)$$
 (2)

To evaluate the internal and external mass transfer resistances, the Biot number (Bi) was calculated for each  $v_s$ , as follows:

$$Bi = \frac{k_s. R_p}{D_e}$$
 (3)

where  $R_p$  is the equivalent sphere radius,  $k_s$  is the liquid-phase mass transfer coefficient and  $D_e$  is the substrate effective diffusivity in the bioparticle.

The values of  $k_sa$  and  $k_s$  were found to be very low, thus indicating that the external mass transfer resistance should be significantly high in immobilized cell reactors operating at low  $v_ss$ , as is expected for HAIS reactors. According to Bailey and Ollis (1986), the effect of external mass transfer resistance is not significant for Bi  $\geq$  100. The values of Bi in this experiment were very low compared to the limit proposed by those authors. Therefore, it can be concluded that the effects of mass transfer resistance on the overall reaction rate due to the low applied  $v_s$  were rather high. Moreover, it can also be seen that  $k_s$ a increased with  $v_s$ , as could be expected, since the increase in  $v_s$  causes a decrease in the thickness of the stagnant liquid layer surrounding the bioparticles.

Therefore, the increase of  $v_s$  leads to the increaseof the external mass transfer rate and, consequently, the increase of the overall process reaction rate. The condition of minimum external mass transfer resistance was not attained in the range of  $v_s$  applied in this experiment since  $k_s$ a has consistently increased with  $v_s$ . Even so, the data permitted the establishment of a relationship between  $k_s$ a and  $v_s$ , as follows:

$$k_s a = -0.244 + 0.271. \exp(1.796. v_s)$$
 (4)

The fitted curve and the experimental data are shown in Figure 6.

These results demonstrated that the external mass transfer resistance can be diminished by increasing  $v_s$ . Therefore, reactor operation at a mass transfer resistance as low as possible depends on the experimental parameters such as limiting  $v_s$ , above which the bioparticles are washed out causing reactor collapse.

#### **Estimation of Intrinsic Kinetic Parameters**

For most biochemical processes, the specific substrate utilization rate (r) has been expressed as a function of substrate concentration (S), as follows:

$$r = r_{\text{max}} \frac{S}{\text{Ks} + S}$$
 (5)

15/03/13 Brazilian Journal of Chemical Engineering - Rational Basis for Designing Horizontal-Flow Anaerobic Immobilized Sludge (HAIS) Reactor for Waste... where r<sub>max</sub> is the maximum specific substrate utilization rate and K<sub>S</sub> is the half velocity coefficient.

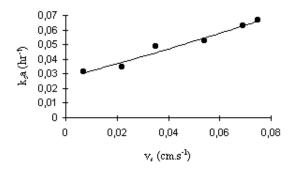


Figure 6: Experimental (• ) and calculated values (— ) of k<sub>s</sub>a from relationship (4).

The estimation of  $r_{max}$  and  $K_S$  followed the sequence below (Zaiat et al., 1996d):

- (i) Values of observed specific substrate utilization rate were correlated with the reciprocal of Bi for different  $S_{\rm h}$ .
- (ii) The specific substrate utilization rate with no liquid-phase mass transfer resistance was obtained by extrapolation when  $Bi^{-1} \rightarrow 0$ .
- (iii) Finally, the intrinsic kinetic parameters ( $r_{max}$  and  $K_S$ ) were estimated by the Hanes-Woolf plot method (Bailey and Ollis, 1986).

The intrinsic kinetic parameters,  $r_{max}$  and  $K_S$ , estimated for a glucose-based substrate and anaerobic sludge immobilized in polyurethane foam matrices at 30° C were found to be 0.330 mg COD/mg VSS.hr and 72 mg COD/l, respectively. Considering first-order kinetics, a value of 1.05 x  $10^{-3}$  l/mg VSS.hr was found for the kinetic parameter.

Another proposed method to estimate intrinsic kinetic parameters was proposed by Vieira et al. (1996). Such a method is based on the minimization of mass transfer resistances by establishment of adequate environmental and operational conditions. The methodology was tested using a glucose-based synthetic wastewater and immobilized anaerobic sludge in polyurethane foam matrices and it was found to provide reliable parameters.

### **Intraparticle Mass Transfer**

Studies on intraparticle mass transfer were carried out by Vela et al. (1996). This work reported a value for effective diffusivity ( $D_e$ ) of 0.66 x  $10^{-5}$  cm<sup>2</sup>/s for glucose in bioparticles of sodium alginate containing anaerobic sludge. This  $D_e$  obtained corresponds to 88% of the value in water. However, this fact can not be extended for other types of bioparticles. Unpublished results from Vela (1996) confirmed that the value of  $D_e$  for low-strength substrates in polyurethane foam matrices containing anaerobic sludge has the value of the diffusion coefficient in water.

# **CONSIDERATIONS ON DESIGN PROCEDURES**

As foreseen by hydrodynamic studies, the HAIS reactor can be simulated considering it an "ideal plug-flow reactor". If first-order kinetics for substrate utilization is assumed, the conservation equation for heterogeneous "plug-flow reactor" can be written as

$$Econ = 1 - \exp\left[\frac{-L \cdot X \cdot k_1 \cdot p}{\mathcal{E}Vs}\right]$$
 (6)

where  $E_{COD}$  is the COD efficiency removal, L is the reactor length, X is the biomass concentration,  $k_1$  is the first-order intrinsic kinetic parameter,  $\epsilon$  is the bed porosity,  $v_s$  is the liquid superficial velocity and  $\eta$  is the global effectiveness factor defined as

$$p = (\frac{3. \text{Bi}}{p^2}). \left[\frac{p (\cosh p - \text{senh } p)}{p (\cosh p + (\text{Bi} - 1). \text{senh } p)}\right]$$
(7)

where  $\phi$  is the Thiele modulus defined as

$$\not = R_{P} \sqrt{\frac{k_{1}.X}{D_{e}}}$$
 (8)

where  $R_p$  is the equivalent sphere radius,  $D_e$  is the substrate effective diffusivity in the bioparticle,  $k_1$  is the first-order intrinsic kinetic parameter and X is the biomass concentration.

Therefore, the HAIS reactor can be designed utilizing equation (6). The use of this equation is possible if the fundamental parameters (intrinsic kinetics and external and internal mass transfer coefficients) are previously determined.

#### **ACKNOWLEDGMENTS**

The authors acknowledge the financial support received for this work from FAPESP-Fundação de Amparo à Pesquisa do Estado de São Paulo for and the grants received from CAPES-Fundação Coordenação de Aperfeiçoamento de Pessoal de Nível Superior and CNPq-Conselho Nacional de Desenvolvimento Científico e Tecnológico.

#### **NOMENCLATURE**

a Interfacial area for mass transfer, cm<sup>-1</sup>

Bi Biot number

D Diameter of the reactor, cm

De Effective diffusivity in the bioparticle, cm<sup>2</sup>/hr

E0 Dimensionless age distribution of fluid

f<sub>VSS</sub> Washed-out VSS accumulated fraction, %

ks Liquid-phase mass transfer coefficient, cm/hr

k<sub>1</sub> First-order kinetic intrinsic parameter, I/mg VSS.hr

K<sub>S</sub> Half-velocity coefficient, mg COD//

L Reactor length, cm

r Specific substrate utilization rate, mg COD/mg VSS.hr

 $r_{\text{max}}$  Maximum specific substrate utilization rate, mg COD/mg VSS.hr

R<sub>D</sub> Equivalent sphere radius, cm

S Substrate concentration, mg COD//

 $S_b$  Substrate concentration in the bulk liquid, mg COD//

 $v_s$  Liquid superficial velocity, cm/s

t Time, hr

X Biomass concentration, mg VSS//

ε Bed porosity

η Effectiveness factor

θ Dimensionless time (t/HDT)

#### **Abbreviations**

COD Chemical oxygen demand

HDT Hydraulic detention time

OLR Organic loading rate

RTD Residence time distribution

TA Total alkalinity

TSS Total suspended solids

VFA Volatile fatty acids

VSS Volatile suspended solids

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