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1.- Introduction

The K_{La} coefficient has been considered as a key parameter to go deeply into the study of the nitrifying fixed bed reactors corresponding to the third compartment of the MELISSA loop. In order to perform an experimental estimation of this mass transfer coefficient, two partners of the MELISSA project have carried out a set of experiments.

In technical note 52.31 the first experiments to determine K_{La} coefficient were carried out with the aid of a developed methodology which includes the use of the liquid flow model in the reactor (previously determined from RTD experiments, TN 37.510).

In the present technical note more experiments have been carried out and the methodology used has been reviewed and slightly modified in order to obtain a more precise approach to the real value of this coefficient in the bed part of the reactor.

The modifications performed are mainly two:

- a) The Henry's constant for the oxygen has been corrected to take into account the ionic strength of the solution;
- b) Experiments in continuous mode have been carried out. This kind of operation using the sulphite method has been used because the parameters of the flow model were obtained with continuous experiments, and the effect of the liquid flow rate would be important.

2.- Description of the methodology used

The methodology used for these experiments is the same already described in TN 52.31, with the only exception of the correction of the Henry's constant to take into account the ionic strength of the sulphite solution. Then the resulting equations are (see figure 1):

$$G_e y_e - G_s y_s = (r_{O_2})_A V_A + (r_{O_2})_B V_B + (r_{O_2})_C V_C \quad (1)$$

$$y_A = \frac{A}{1+A}; \quad \text{where, } A = \frac{y_e - (r_{O_2})_A \frac{V_A}{G_e}}{1 - y_e} \quad (2 \text{ and } 3)$$

$$y_B = \frac{A}{1+A}; \quad \text{where, } A = \frac{y_A - (r_{O_2})_B \frac{V_B}{G_A}}{1 - y_A}; \quad \text{with, } G_A = G_e - (r_{O_2})_A V_A \quad (4, 5 \text{ and } 6)$$

$$y_s = \frac{A}{1+A}; \quad \text{where, } A = \frac{y_B - (r_{O_2})_C \frac{V_C}{G_B}}{1 - y_B}; \quad \text{with, } G_B = G_A - (r_{O_2})_B V_B \quad (7, 8 \text{ and } 9)$$

$$C_A^* = \frac{y_e P_e - y_A P_A}{H \ln \frac{y_e P_e}{y_C P_C}} \quad (10)$$

$$(k_L a)_A = \frac{(r_{O_2})_A}{C_A^*} \quad (11)$$

$$C_B^* = \frac{y_A P_A - y_B P_B}{H \ln \frac{y_A P_A}{y_B P_B}} \quad (12)$$

$$(k_L a)_B = \frac{(r_{O_2})_B}{C_B^*} \quad (13)$$

$$C_C^* = \frac{y_B P_B - y_C P_C}{H \ln \frac{y_B P_B}{y_C P_C}} \quad (14)$$

$$(k_L a)_C = \frac{(r_{O_2})_C}{C_C^*} \quad (15)$$

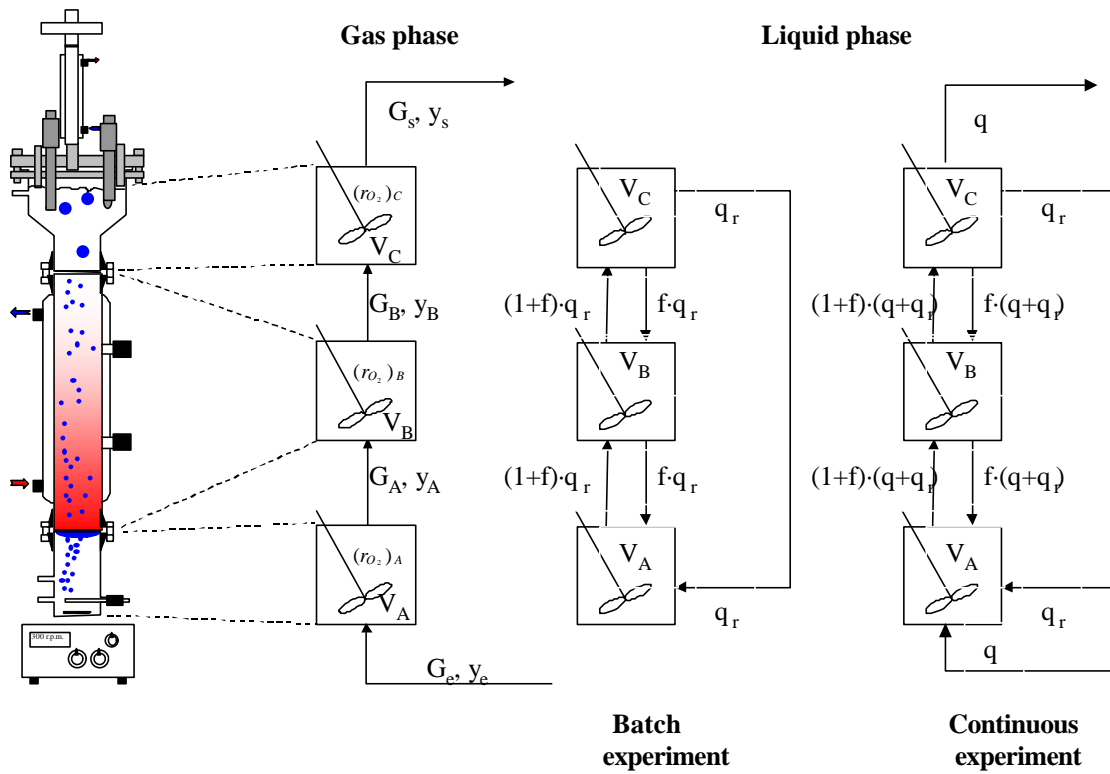


Figure 1.- Nomenclature used in the oxygen balances to apply the sulphite method. Where y_i are the oxygen molar fractions in the gas flow, of total flow-rate of G_b , V_i is the liquid volume in each part (A, B, C), and $(r_{O_2})_i$ is the oxygen consumption rate (oxygen moles per time and volume unit) in each section of the reactor (A, B, C). A diagram with the terminology used regarding the mass balances for liquid phase is also detailed; where q_r is the recirculation flow-rate, q the feed flow rate and f the backmixing coefficient.

Henry's constant (H in Pa·L/mol; H_e in Pa) has been corrected taking into account the ionic strength of the solution used in the experiments. From the model developed by Schumpe *et al.* (1982) to compute the oxygen solubility in culture mediums, it is possible to determine the influence of the electrolyte concentration in the oxygen solubility:

$$\log \frac{H_e}{H_{e_B}} = \sum H_i I_i \quad (16)$$

Where He_B is the Henry's constant (Pa) in pure water (tabled as a function of temperature), H_i are the model parameters specific for each ion at a given temperature. I_i is the ionic strength of each ion, and is calculated as follows:

$$I_i = \frac{1}{2} c_i z_i^2 \quad (17)$$

Where c_i is the concentration of a particular ion (mol/L) and z_i is the ion charge as an absolute value.

When oxygen concentration is decreasing in gas phase, the total gas flow-rate consequently, also decreases. On the other hand, the oxygen transferred to liquid phase reacts with sulphite to produce sulphate. In that way, the sulphite concentration in each of the three parts of the reactor (A, B and C), varies due to two reasons: the reaction with transferred oxygen from gas phase and, on the other hand, due to the mix of the liquid phase. The mass balances corresponding to sulphite in each part of the reactor can be established from the flow model already described (TN 52.31).

3.- Batch experiments

If the experiments are carried out working in batch, the sulphite mass balances are (see also figure 1):

$$\frac{dC_A}{dt} = \frac{1}{V_A} [q_r \cdot C_C + f \cdot q_r \cdot C_B - (1+f) \cdot q_r \cdot C_A] - 2 \cdot (r_{O_2})_A \quad (18)$$

$$\frac{dC_B}{dt} = \frac{1}{V_B} [(1+f) \cdot q_r \cdot C_A + f \cdot q_r \cdot C_C - (1+2f) \cdot q_r \cdot C_B] - 2 \cdot (r_{O_2})_B \quad (20)$$

$$\frac{dC_C}{dt} = \frac{1}{V_C} [(1+f) \cdot q_r \cdot C_B - (1+f) \cdot q_r \cdot C_C] - 2 \cdot (r_{O_2})_C \quad (21)$$

$$\text{Con } (r_{SO_3^{2-}})_i = 2 \cdot (r_{O_2})_i; \quad i = A, B, C \quad (22)$$

During experiments the sulphite concentration has been determined at each instant t , in each one of the parts of the reactor (C_A , C_B , C_C). The value of the variables V_i , q_r , f y V_m is known in the conditions in which the experiment is carried out. Therefore, is possible to build an objective function that allows to fit, using least squares method, the theoretical evolution of the sulphite concentrations in each tank to the experimental evolution of these concentrations, determined through titration of the withdrawn samples. This optimisation is carried out using as adjustable parameters the unknowns of the last set of ordinary differential equations: $(r_{O_2})_i$. Using this values $(r_{O_2})_i$ is possible to estimate the K_La coefficient in each part of the reactor using the equations 1-15.

The objective function used is presented in equation 23, which is a non linear weighted least square function, in which a major weight is imposed to the values corresponding to the bed (part B), because a major precision is necessary to this part (in the bed the biological degradation is produced). In this equation, $y_{i,j}^x$ are the theoretical ($x = theoretical$), and experimental ($x = experimental$) concentrations of the sections A, B and C (for $j = A, B, C$), at different time instants t , from $i = 1, 2, 3, \dots, n$, where n is the number of experimental points.

$$f_{objective} = 0.25 \sum_{i=1}^n (y_{i,A}^{theor.} - y_{i,A}^{experimental})^2 + 0.5 \sum_{i=1}^n (y_{i,B}^{theor.} - y_{i,B}^{experimental})^2 + 0.25 \sum_{i=1}^n (y_{i,C}^{theor.} - y_{i,C}^{experimental})^2 \quad (23)$$

In figure 24 the calculation diagram of the optimization procedure is described. All computations have been carried out using MATLAB (v. 4.2b, The MathWorks, Inc. 1994).

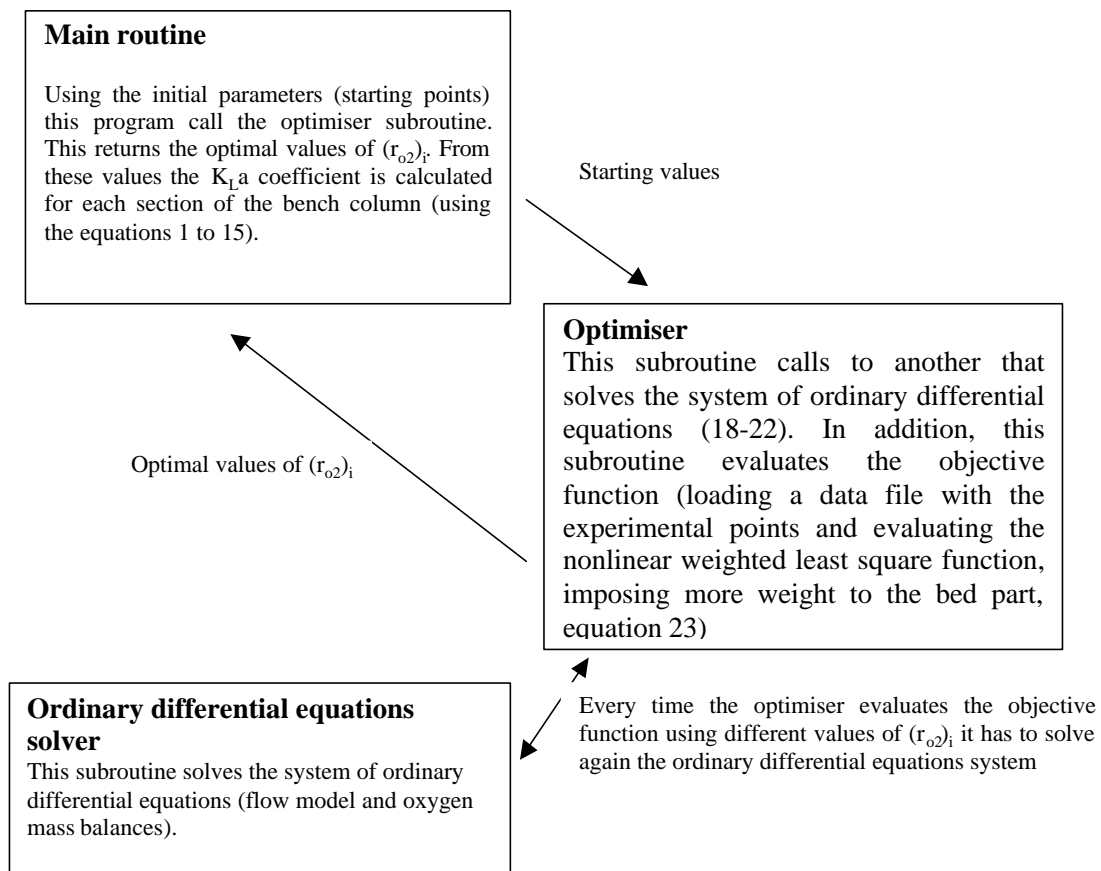


Figure 2.- Structure and functionality of the main routines used in the optimization of the batch experiments of determination of $K_L a$ coefficient using a chemical method.

Experimental procedure

The reactor with any liquid inside is filled under inert atmosphere (nitrogen), with a sodium sulphite solution (50-100 g/L). At time zero the aeration is switched on and samples are withdrawn from the three parts of the reactor (see figure 3) under inert atmosphere. This samples are added to a solution containing I_2 (25.38 g/L) and KI (70 g/L). After titration with thiosulphate, the sulphite concentration as function of time corresponding to the different sections of the reactor is calculated with equation 24. All experiments have been carried out at 30 °C.

$$C_{SO_3^{2-}} = \frac{C_{I_2} V_{I_2} - 0.5 \cdot C_{S_2O_3^{2-}} V_{S_2O_3^{2-}}}{V} \quad (24)$$

Where $C_{SO_3^{2-}}$ is the sulphite concentration at time t ; C_{I_2}, V_{I_2} are the concentration and the volume corresponding to the solution used to add the samples withdrawn from the reactor; $C_{S_2O_3^{2-}}$ is the thiosulphate concentration used for titration; $V_{S_2O_3^{2-}}$ is the volume required in a titration and, V is the volume of the sample.

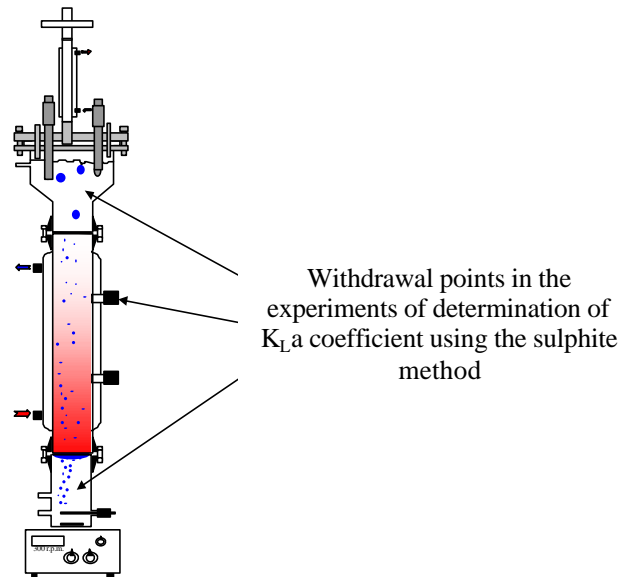


Figure 3.- Points of sample withdrawal in the bench nitrifying reactor during the K_La experiments using the sulphite method.

Experimental results

A new experiment has been carried out using the sulphite method with an aeration flow-rate of 40 mL/min and without liquid recirculation. The results after optimization are shown in figure 4 and in table 1. In addition, a study to determine the precision of the fitted parameters in the optimization has been carried out (figure 5).

As can be observed in figure 4, the evolution of the sulphite concentration in each part of the reactor is different. The variation in the sulphite concentration in the experiment is due to two different reasons: firstly, the oxygen transfer from the gas to the liquid phase, and secondly, the mixing of the liquid phase. The mixing of the liquid phase between sections is produced mainly due to the effect of the gas bubbles in the liquid phase (in this experiment is carried out without external liquid recirculation).

Although the fitted results obtained are satisfactory, the sensitivity analysis carried out shows that the precision associated with the K_La coefficient associated to bed is higher than in the case of the two coefficients corresponding to both, bottom and top parts of the reactor. On the other hand, a clear drawback of this type of determination is that the flow model parameters could not be accurate enough due to the fact that the RTD experiments were carried out in continuous mode. For this reason, a new experiment were performed in continuous mode, to operate strictly using the same conditions than those used in RTD experiments.

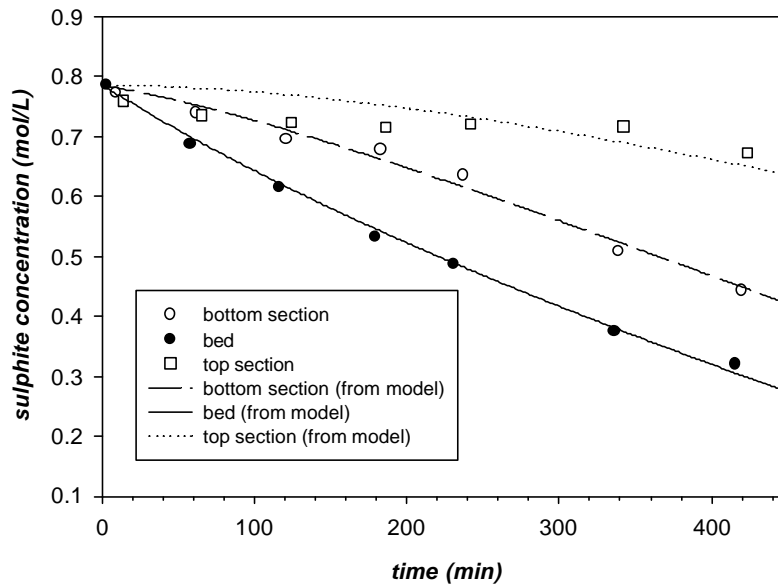


Figure 4.- Determination of the $K_L a$ coefficient using the sulphite method. Batch experiment.

Table 1.- Results obtained in the determination of the $K_L a$ coefficient in each part of the reactor sections, as can be observed in figure 4.

Reactor section	$K_L a$ (s^{-1})
Bottom	0.026
Bed	0.13
Top	0.002

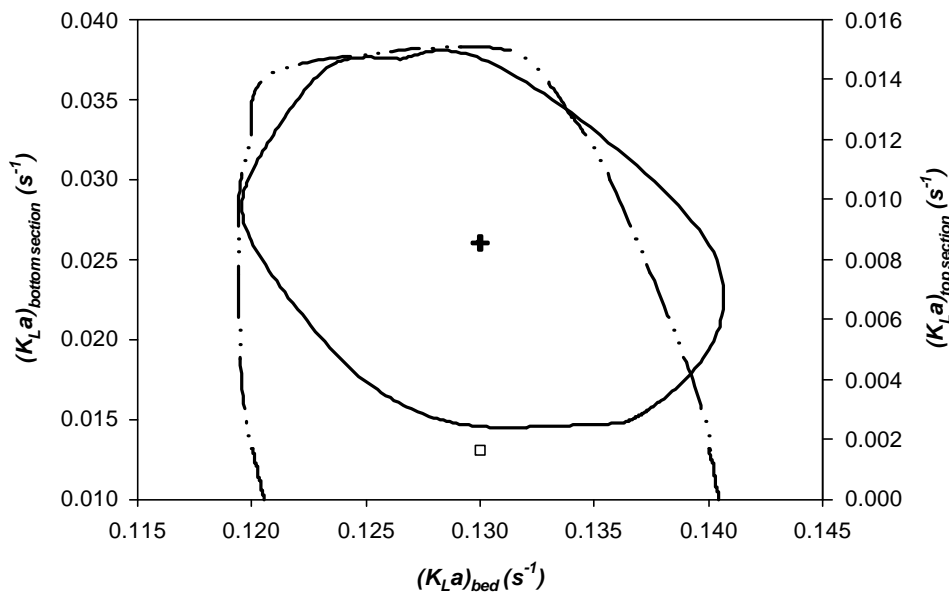


Figure 5.- Estimation of the confidence contours using a 95 % confidence level.

— Confidence contour corresponding to bed and bottom $K_L a$ coefficients (using a value for the bottom $K_L a$ coefficient of $0.02 s^{-1}$ (+ optimal values of the parameters).

— — — Confidence contour corresponding to bed and top $K_L a$ coefficients (using a value for the bottom $K_L a$ coefficient of $0.03 s^{-1}$ (\square optimal values of the parameters).

4.- Experiments operating in continuous mode

The reactor operates in continuous mode (using distilled water as feed), the sulphite balances are (see also figure 1):

$$\frac{dC_A}{dt} = \frac{1}{V_A} [q_r \cdot C_C + f \cdot (q + q_r) \cdot C_B - (1 + f) \cdot (q + q_r) \cdot C_A] - 2 \cdot (r_{O_2})_A \quad (25)$$

$$\frac{dC_B}{dt} = \frac{1}{V_B} [(1 + f) \cdot (q + q_r) \cdot C_A + f \cdot (q + q_r) \cdot C_C - (1 + 2 \cdot f) \cdot (q + q_r) \cdot C_B] - 2 \cdot (r_{O_2})_B \quad (26)$$

$$\frac{dC_C}{dt} = \frac{1}{V_C} [(1 + f) \cdot (q + q_r) \cdot C_B - (1 + f) \cdot (q + q_r) \cdot C_C] - 2 \cdot (r_{O_2})_C \quad (27)$$

$$\text{With } (r_{SO_3^{2-}})_i = 2 \cdot (r_{O_2})_i; \quad i = A, B, C \quad (28)$$

The computation diagram used is exactly the same described in figure 2, but using equations 25 to 28.

The experimental procedure used has been basically the same already described, but using a higher sulphite concentration (100-150 g/L, because the experiment is carried out in continuous mode and for this reason the decrease in sulphite concentration is more important). The distilled water used as inlet of the reactor is maintained under inert atmosphere by a constant nitrogen bubbling.

An experiment using 40 mL/min as aeration flow-rate and a recirculation flow-rate of 4.5 mL/min (the temperature of the experiment was 30 °C) has been carried out. The results obtained after optimization are shown in figure 6 and in table 2. In addition, a study to determine the precision of the fitted parameters in the optimization has been carried out (figure 7).

Table 2.- Results obtained in the determination of the K_La coefficient in each part of the reactor sections, as can be observed in figure 6.

Reactor section	K_La (s ⁻¹)
Bottom	0.024
Bed	0.077
Top	0.002

Using exactly the same conditions described in this experiments a RTD experiment had been carried out (see TN 37.510), for this reason the flow model parameters are perfectly known in this operating conditions.

On the other hand, a key drawback can be observed in figures 6 and 7: in the continuous mode experiment, the decrease of the sulphite concentration in the different parts of the reactor is very important, and this fact produces a decrease in the sensitivity of the fitted parameters.

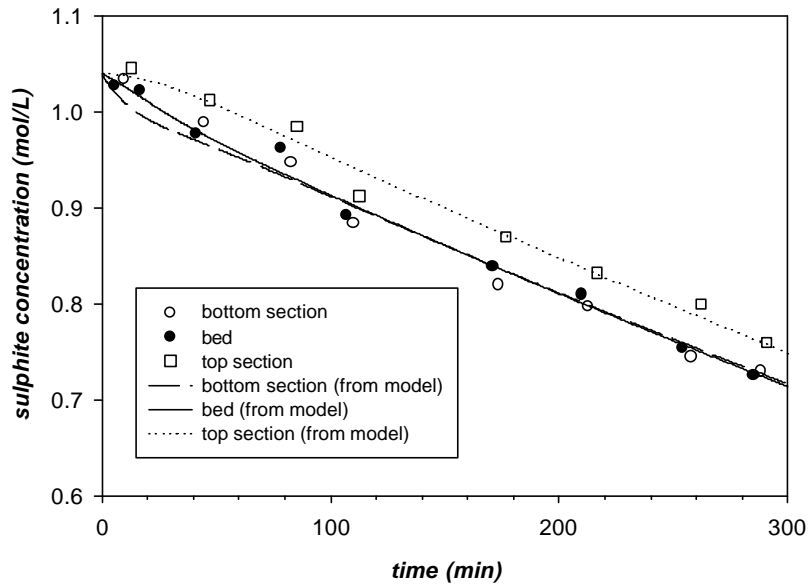


Figure 6.- Determination of the $K_L a$ coefficient using the sulphite method. Continuous experiment.

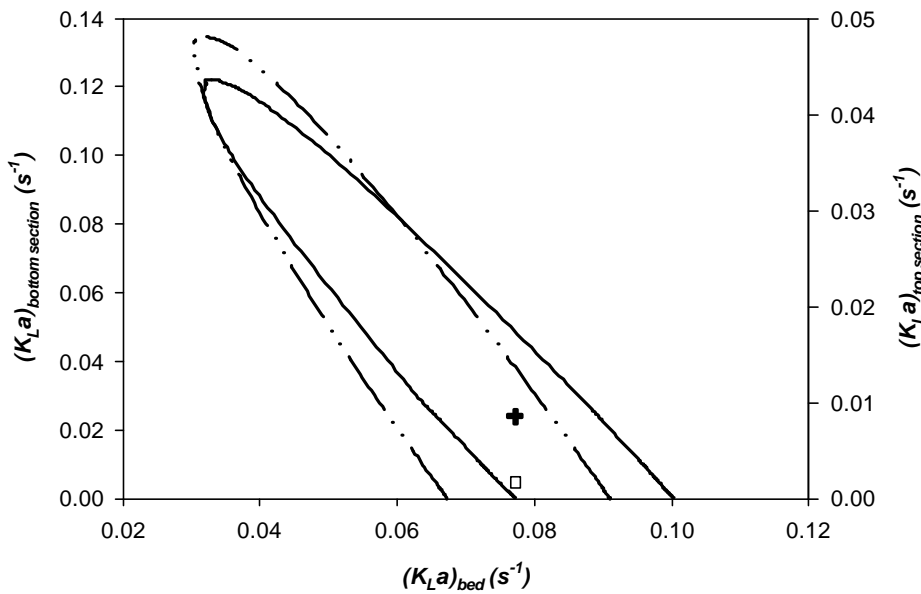


Figure 7.- Estimation of the confidence contours using a 95 % confidence level.
 — Confidence contour corresponding to bed and bottom $K_L a$ coefficients (using a value for the bottom $K_L a$ coefficient of 0.002 s^{-1} (+ optimal values of the parameters).
 — — — Confidence contour corresponding to bed and top $K_L a$ coefficients (using a value for the bottom $K_L a$ coefficient of 0.02 s^{-1} (\square optimal values of the parameters).

5.- Conclusions

The results obtained with this set of experiments demonstrate that K_La coefficient has a strong variation along the reactor. The results obtained are therefore, a first approach to the real value of the K_La coefficient in the bed, although the best way to determine the coefficient is to perform an estimation during the operation of the reactor, as it is described in next section.

Nevertheless, the sulphite method has some drawbacks. First of all, the sulphite solution used during the experiments can not approach the properties of the culture broth (with variable composition when operating conditions change). In addition, the concentrations used during the experiments have enough ionic strength as to impact in the interfacial area and even in the mass transfer coefficient (van't Riet, 1979). On the other hand, the reaction is assumed to be a zero order in respect to the sulphite concentration, but the reaction is not sufficiently fast, and for this reason it takes place in the bubble surrounding film. This fact generates a decrease in the mass transfer coefficient and overestimation of the volumetric mass transfer coefficient (Blanch and Clark, 1996).

6.- Estimation of the K_La coefficient during the operation of the reactors

In literature partial nitrification over nitrite has been widely described (Hanaki *et al.*, 1990; Garrido, 1997; Nogueira, 1998; Campos, 2000). The dissolved oxygen concentration that produces the nitrite accumulation in biofilm nitrification processes is around 2.5 ppm of dissolved oxygen (Garrido, 1997; Joo *et al.*, 2000). If a range of 0-2.5 ppm of dissolved oxygen concentration is assumed, is possible to estimate the oxygen volumetric transfer, K_La using a direct method (Kargi and Moo-Young, 1985; equation 29), in the experiments in which oxygen supply become limiting. In the following sections the results obtained using this method in the two nitrifying reactors are discussed.

$$K_La = \frac{Y_{NO_2^-} \cdot r_{NO_2^-} + Y_{NO_3^-} \cdot r_{NO_3^-}}{(C^* - C)} \quad (29)$$

Where Y_i are the theoretical stoichiometric coefficients from ammonium oxidation over nitrite (1.5 mol O_2 /mol NO_2^-) and the from ammonium over nitrate (2mol O_2 /mol NO_3^-), r_i are the nitrite and nitrate flow in the effluent; C^* is the dissolved oxygen concentration of saturation and ICI the dissolved oxygen concentration in steady state (that can be assumed in the range 0-2.5 as already discussed above).

6.1.- Bench columns

The results obtained form the experiments already performed and discussed in TN 37.520, the results are detailed in table 3.

Table 3.- Estimation of the K_La coefficient in the bed (bench columns) using a direct method in the experiments with limiting oxygen supply.

Aeration flow-rate (mL/min)	Ammonium input load (kg N· m ⁻³ · día ⁻¹)	Ammonium removed load (%)	Total removed load (%)	$\frac{mol O_2}{mol NH_4^+}$	K_La range (s ⁻¹) (direct method)
9	0.42	85.3	82.7	6.3	[1.4 – 2.0]· 10 ⁻²
15	0.42	91.6	89.9	10.5	[1.5 – 2.1]· 10 ⁻²
9	0.55	85.3	77.7	4.7	[1.8 – 2.6]· 10 ⁻²
40	1.11	83.3	68.2	10.5	[2.1 – 3.1]· 10 ⁻²

The dissolved oxygen concentration of saturation has been determined from the Henry's constant modified by the ionic strength of the solution (using the model described by Schumpe *et al.* already detailed in the present technical note, equations 16 and 17). In addition, the concentration of saturation has been corrected to take into account the effect of the pressure using the following expression (Sekizawa *et al.*, 1985):

$$C_{correctada}^* = C^* \left[1 + \frac{\mathbf{r}g}{P_{atm}} \frac{1 - \mathbf{e}_g}{1 - \mathbf{e}_p} (h - l) \right] \quad (30)$$

Where \mathbf{r} is the liquid density (kg/m^3), g the gravity acceleration (m/s^2), P_{atm} the atmospheric pressure (Pa), \mathbf{e}_g the gas fraction, \mathbf{e}_p the solid fraction in the reactor, h the sparger depth and l the length from the sparger, both in meters.

Fujie *et al.* (1992) studied the volumetric transfer coefficient in a fix bed bioreactor and determined the value of the coefficient as a function of the biofilm concentration in the reactor. The results demonstrated that the K_La coefficient decreased when the biofilm concentration increases in the reactor. The K_La value is 2 times lower than the value before inoculation of the bioreactor.

If the values shown in table 3 are compared with the results from sulphite experiments (table 2), it is possible to conclude that the value of the coefficient without biofilm (0.077 s^{-1}) is also higher in our case, than the one obtained with mature biofilm (0.026 s^{-1}).

When liquid superficial velocity is lower than the one corresponding to the gas, a correlation with the following expression is commonly used (Carberry and Varma, 1986):

$$K_La = a \cdot u_G^b \quad (31)$$

Where a and b are parameters and u_G is the gas superficial velocity. If the different values from the estimation of the coefficient are correlated with the gas superficial velocity, the following expression is obtained (see also figure 8):

$$K_La = 0.082 \cdot u_G^{0.35} \quad (32)$$

With u_G in cm/s and K_La in s^{-1} .

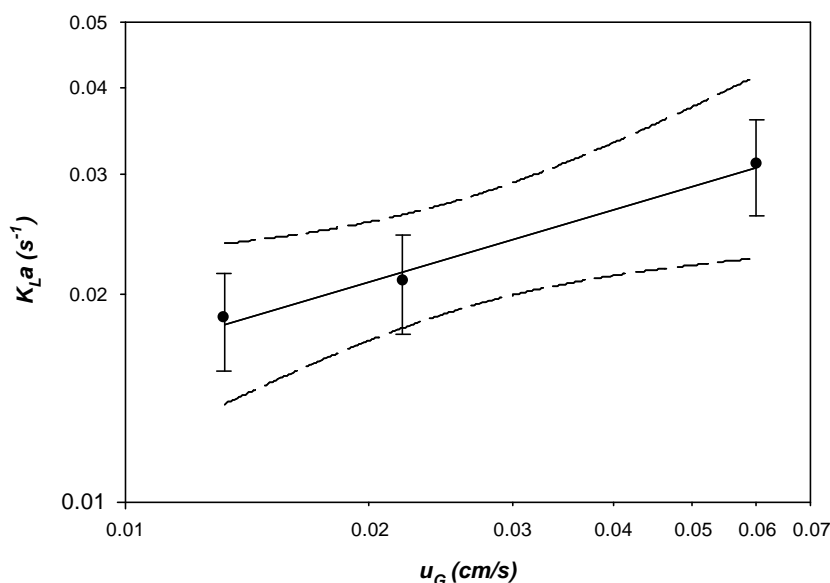


Figure 8.- Correlation between the K_{La} coefficient and the gas superficial velocity. The dashed line (— —) is the precision of the fitting using a confidence level of 95 %.

6.2.- Pilot reactor

The same procedure already discussed to determine the K_{La} coefficient using a direct method in the bench columns has been carried out also in the pilot reactor. The experiments in which the oxygen supply become limiting in the pilot reactor has been already described in TN 43.43. Nevertheless, in the case of the pilot reactor there is an important difference regarding this topic: the aeration flow-rate is not used as an operating condition, and therefore remains constant. In that way, to control the oxygen supply in the reactor the oxygen partial pressure is manipulated with the oxygen and nitrogen mass flow-meters.

The dissolved oxygen concentration in the bottom and top parts of the pilot reactor is known (where the dissolved oxygen probes are placed). On the contrary, the dissolved oxygen concentration in the bed is not known in the bed. The measured oxygen concentration can be assumed as the concentration of saturation, because in the top and bottom parts of the reactor there is no biomass, and consequently the oxygen consumption can be neglected.

In order to estimate the K_{La} coefficient in the bed using the direct method already described is necessary to fix a dissolved oxygen concentration in the bed. In the already discussed study performed by Garrido *et al.* (1997), the oxygen was limiting at 2.5 mg/L and the ammonium oxidation was completed (only nitrite accumulation) if the dissolved oxygen concentration was higher than 1.8 mg/L. Assuming this range the estimation of the K_{La} coefficient can be performed. Results are presented in table 4 (in the shadowed cells).

Once the value of the coefficient is estimated, this can be used to determine the dissolved oxygen concentration in the bed in the rest of steady states in which the oxygen became limiting. The value of the coefficient can be considered as constant because the aeration flow-rate is kept constant (3 L/min).

Table 4.- Determination of the K_La coefficient (shadowed cells, bold). Using this value (assumed as constant in all the experiments) the dissolved oxygen concentration in the bed can be calculated in the rest of experiments.

Dissolved oxygen concentration (from probes %)	Removed ammonium load (%)	Total removed load (%)	Concentration of saturation (mg/L)	Dissolved oxygen concentration in the bed (mg/L)	K_La (s^{-1})
40	68.5	64.8	3.5	[2.3-2.8]	[0.25-0.15]
20	96.0	34.3	1.8	[0.1-0.8]	[0.25-0.15]
40	99.8	61.6	3.5	[1.8-2.5]	[0.25-0.15]
80	99.8	99.9	7.0	[5.2-5.9]	[0.25-0.15]

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