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1.- SULFITE METHOD

1.1.- Principle

The Cooper's method allows to determine the oxygen consumption rate by titration. To carry out this method it is necessary to fill the bioreactor with a sodium sulfite solution and to evaluate, as function of time, the decrease of the sodium sulfite concentration. The reaction that takes place is as follows:

$$SO_3^{2-} + \frac{1}{2} O_2 \rightarrow SO_4^{2-}$$

Using certain conditions it can be demonstrated that the order of this reaction is zero (in respect to the sodium sulfite), hence, the sulfite oxidation rate is maintained constant and equal to twice the oxygen consumption rate. Therefore, the evolution of the sulfite concentration with time is linear decreasing.

On the other hand, the oxygen mass balance in the bioreactor, assuming liquid and gas phases perfectly mixed, is:

$$\frac{dC}{dt} = K_L a (C^* - C) - r_{O_2} \quad ;$$

being:

r_{O2} = oxygen consumption rate,

C = oxygen concentration in the liquid phase

C^* = oxygen concentration in the liquid phase at equilibrium conditions

K_La = oxygen transfer coefficient

Considering the low solubility of the oxygen in the culture medium, it is possible to assume that all oxygen transferred to the liquid phase is consumed instantaneously and so dC/dt = 0, thus the following equation applies:

$$r_{O_2} = K_L a (C^* - C)$$

The reaction described is catalysed by cobalt ions. Using this catalyst (in concentration lower than 10^{-8} mol/L) it is possible to assume that all the dissolved oxygen is consumed. Hence:

$$C = 0$$

Using this information, the K_La can be expressed as:

$$K_La = \frac{r_{O_2}}{C^*}$$

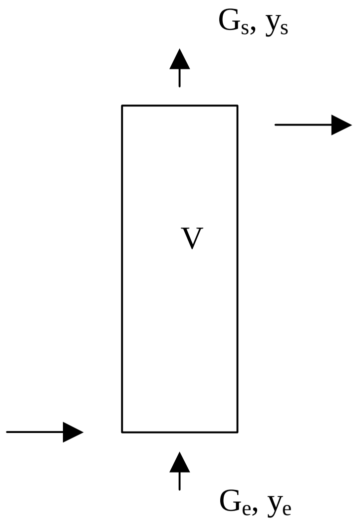
The oxygen mass balance in the bioreactor and the expression of the saturation concentration for the oxygen are:

$$G_e y_e - G_s y_s = r_{O_2} V$$

$$y_s = \frac{A}{1+A};\text{where, } A = \frac{y_e - r_{O_2}\dfrac{V}{G_e}}{1 \quad y_e}$$

$$C^* = \frac{y_{O_2} P_t}{H};\text{where, } y_{O_2} = \frac{y_e - y_s}{\ln \dfrac{y_e}{y_s}}$$

$$K_La = \frac{r_{O_2}}{C^*}$$



Where:

V = reactor volume

y_e = oxygen molar fraction in the gas phase in the input flow to the bioreactor

y_s = oxygen molar fraction in the gas phase in the output flow to the bioreactor

G_e = input gas flow-rate

G_s = output gas flow-rate

H = Henry's constant

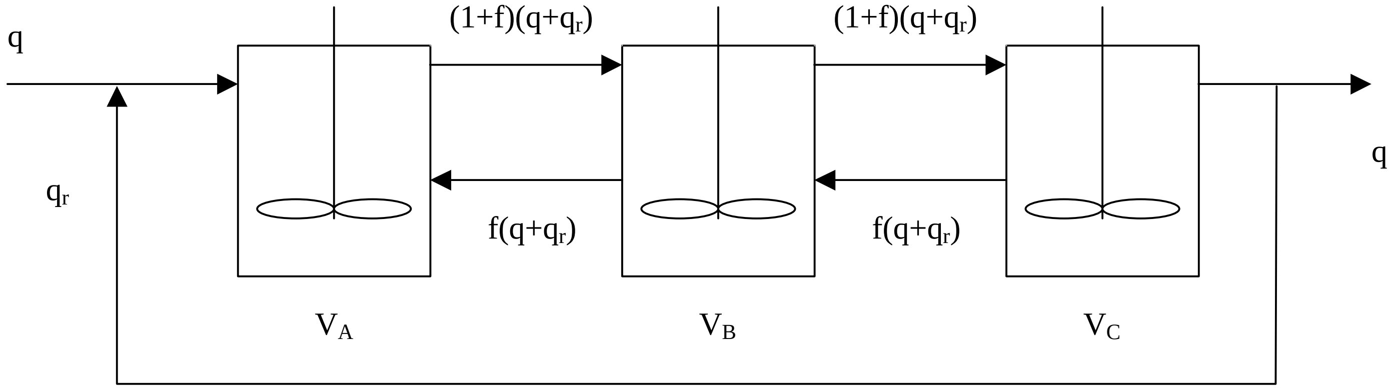
P_t = total pressure in the bioreactor

The oxygen consumption rate (r_{O2}) is determined from the titration of different samples withdrawn from the reactor as a function of time.

This detailed procedure is the classical or standard protocol to determine the K_La coefficient, it has been developed for well mixed reactor, and therefore it is not possible to use it in the case of the bench columns, otherwise an important mistake at the time of estimating the oxygen consumption rate (r_{O2}) would be made. First, the K_La along the height of the column is expected to be non-homogeneous, due to geometry of the different sections of the reactor. And, on the other hand, consequently, the sulfite concentration of each part of the bench column will influence on the others due to the effect of the liquid phase mixing in the reactor. Therefore, it is absolutely essential to include the effect of the liquid phase mixing in the determination of the K_La coefficient.

In this way, the flow model of the bench column has to be perfectly known at working conditions. For this reason, the next point in this report is the study of the flow model to use in the numerical calculations of the K_La determination.

1.2.- Flow model



The model of the bench column has been structured considering the reactor divided in three different parts: bottom (A), bed (B) and top (C) that are well mixed tanks (see **figure 3** in point 1.4. of this document). Moreover, a recirculation flow has been added (in fact, this external recirculation flow-rate already exits physically) and an internal back-mixing.

Flow model (ordinary differential equations from tracer mass balances), constants and initial values:

$$\frac{dC_A}{dt} = \frac{1}{V_A}\left[q_r \cdot C_C + f \cdot (q + q_r) \cdot C_B - (1 + f)(q + q_r) \cdot C_A\right] \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

$$\frac{dC_B}{dt} = \frac{1}{V_B}\left[(1 + f)(q + q_r) \cdot C_A + f \cdot (q + q_r) \cdot C_C - (1 + 2 \cdot f)(q + q_r) \cdot C_B\right] \ldots\ldots\ldots\ldots (2)$$

$$\frac{dC_C}{dt} = \frac{1}{V_C}\left[(1 + f)(q + q_r) \cdot C_B - (1 + f)(q + q_r) \cdot C_C\right] \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

With : $C_A = \dfrac{M}{V_A}$ when time is equal to zero;

$C_B = C_C = 0$

For the bench scale columns

$V_A = 0.086$ L

$V_B = 0.164$ L

$V_C = 0.214 \quad V_{dead}$ L

$q = [0.80 - 0.85] \cdot 10^{-3}$ L / min (liquid fed flow - rate to the system)

$q_r = [4.5, 0] \cdot 10^{-3}$ L / min (liquid recycling flow - rate)

Where : V_i = volume of liquid of part i; V_{dead} = dead volume of part B; and

f = back - mixing coefficient; M = amount of tracer (g)

The back-mixing coefficient is one of the fitting parameters used. This parameter is clearly linked to the aeration flow-rate. On the other hand, V_{dead} is the dead volume of the top part (C) and it is the other fitting parameter used.

The data of RTD experiments performed in the packed-bed (TN 37.510) were fitted to the flow model proposed, in the experimental conditions corresponding to those of the sulfite method. From these equations, the residence time distribution experiments carried-out in the bench columns (TN 37.510) allow to fit this flow model in the conditions used in the sulfite method experiment. In this way, it is possible to determine these two parameters for these working conditions: the back-mixing coefficient and the dead volume of liquid of the top part of the bench column.

Results of the fitting of the flow model in the bench columns

Experiment 3 (recirculation flow-rate: 4.5 mL/min; aeration flow-rate: 40 mL/min; input liquid flow-rate q = 0.8544 mL/min). Experimental data in **figure 1**.

*Results of the optimisation (see **figure 1**):*

Back-mixing coefficient fixed: 0.1370; back-mixing flow-rate = 0.7 mL/min
Optimal dead volume estimated: 0.0444 L.
Confidence regions of the estimated parameter:

Confidence level 95 %	$0.0384 \leq V_{dead}$ (L) ≤ 0.0504
Confidence level 99 %	$0.0361 \leq V_{dead}$ (L) ≤ 0.0523

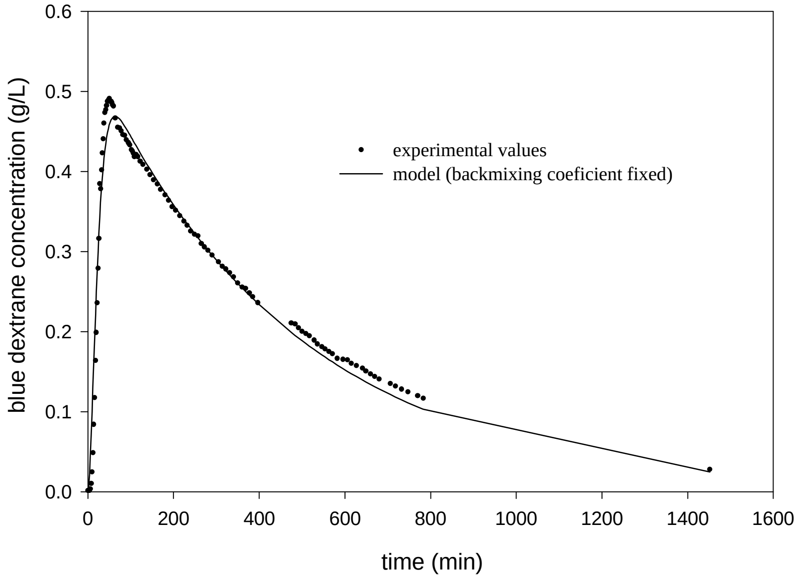
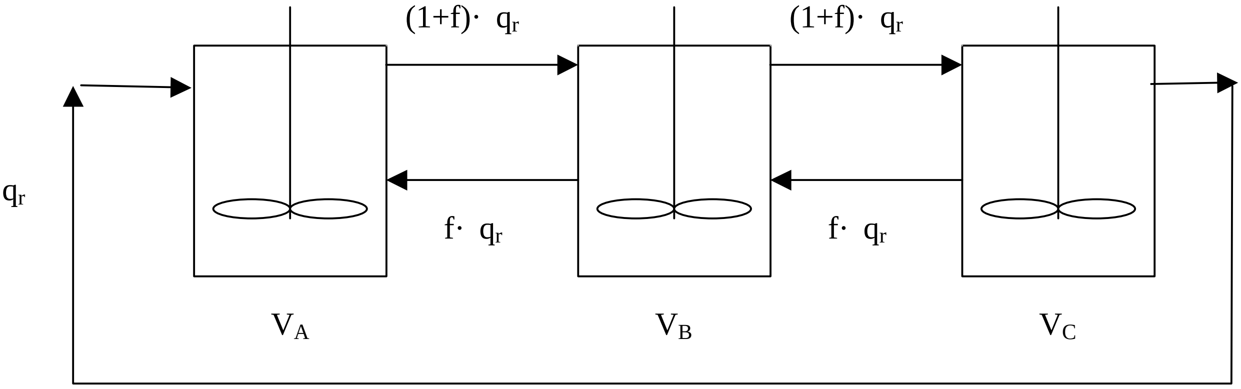


Figure 1 .- Experimental data from RTD experiment (corresponding to figure 13 of TN 37.510) and numerical approach after optimisation.

1.3.- Flow model applied to the experiment of determination of the K_La coefficient using the sulfite method

Once the parameters of the flow model have been determined, it is possible to write the corresponding mass balances for sulfite in each tank:



Mass balances for the sulfite, constants and initial values:

$$With\, q = 0\, and \left(r_{SO_3^{2-}}\right)_i = 2\cdot\left(r_{O_2}\right)_i$$

$$\begin{cases} \dfrac{dC_A}{dt} = \dfrac{1}{V_A}\left[q_r\cdot C_C + f\cdot q_r\cdot C_B - (1+f)\cdot q_r\cdot C_A\right] - 2\cdot(r_{O_2})_A \text{.............................(4)} \\ \dfrac{dC_B}{dt} = \dfrac{1}{V_B}\left[(1+f)\cdot q_r\cdot C_A + f\cdot q_r\cdot C_C - (1+2\cdot f)\cdot q_r\cdot C_B\right] - 2\cdot(r_{O_2})_B \text{.................(5)} \\ \dfrac{dC_C}{dt} = \dfrac{1}{V_C}\left[(1+f)\cdot q_r\cdot C_B - (1+f)\cdot q_r\cdot C_C\right] - 2\cdot(r_{O_2})_C \text{....................................(6)} \end{cases}$$

$$With: \quad C_A = C_B = C_C = 0.4007\, mol/L \qquad when\, time\, is\, equal\, to\, zero;$$

$$V_A = 0.086\, L$$
$$V_B = 0.164\, L$$
$$V_C = 0.2392 - V_{dead}\; L = 0.1948\, L$$
$$q_r = 4.5\cdot 10^{-3}\, L/min$$
$$\text{Back} - \text{mixing coefficient,}\quad \text{f} = 0.1370$$

Where $(r_{o2})_i$ is the oxygen flow (mol/L· min) that is reacting with sulfite (zero order reaction when the sulfite concentration is higher than 0.16 mol/L) in the tank i. This oxygen consumption rate is half of the sulfite consumption rate (stoicheometry).

In this way, from this oxygen consumption rates (that is to say, mass transfer rates, due to the fact that the sulfite method assumes that the oxygen concentration in the liquid phase is zero), it is possible to calculate the K_La coefficient from the mass balances detailed in equations 7 to 24.

$$G_e y_e - G_s y_s = (r_{O_2})_A V_A + (r_{O_2})_B V_B + (r_{O_2})_C V_C \quad (7)$$

$$y_A = \frac{A}{1+A}; \text{where}, A = \frac{y_e - (r_{O_2})_A \frac{V_A}{G_e}}{1-y_e} \quad (8),(9)$$

$$y_B = \frac{A}{1+A}; \text{where}, A = \frac{y_A - (r_{O_2})_B \frac{V_B}{G_A}}{1-y_A}; \text{with}, G_A = G_e - (r_{O_2})_A V_A \quad (10),(11),(12)$$

$$y_s = \frac{A}{1+A}; \text{where}, A = \frac{y_B - (r_{O_2})_C \frac{V_C}{G_B}}{1-y_B}; \text{with}, G_B = G_A - (r_{O_2})_B V_B \quad (13),(14),(15)$$

$$C_A^* = \frac{(y_{O_2})_A P_t}{H}; \text{where}, (y_{O_2})_A = \frac{y_e - y_A}{\ln \frac{y_e}{y_A}} \quad (16),(17)$$

$$(K_La)_A = \frac{(r_{O_2})_A}{C_A^*} \quad (18)$$

$$C_B^* = \frac{(y_{O_2})_B P_t}{H}; \text{where}, (y_{O_2})_B = \frac{y_A - y_B}{\ln \frac{y_A}{y_B}} \quad (19),(20)$$

$$(K_La)_B = \frac{(r_{O_2})_B}{C_B^*} \quad (21)$$

$$C_C^* = \frac{(y_{O_2})_C P_t}{H}; \text{where}, (y_{O_2})_C = \frac{y_B - y_s}{\ln \frac{y_B}{y_s}} \quad (22),(23)$$

$$(K_La)_C = \frac{(r_{O_2})_C}{C_C^*} \quad (24)$$

1.4.- Experimental data and working conditions of the experiment

The operating conditions used to carried-out the experiment are detailed in **table 1.** The samples were withdrawn using an inert atmosphere and the points of sampling are specified in **figure 3**.

By titration of samples, the sulfite concentration is determined at different times, and therefore, it is possible to know the evolution of the sulfite concentration in the different sections of the bench column. These experimental evolutions of the sulfite concentration in the different sections of the reactor are presented in **table 2**.

Stirring rate	300 r.p.m.
Aeration flow-rate	40 mL/min
Recirculation flow-rate	4.5 mL/min
Temperature	30 °C

Table 1.- Operating conditions of the experiment

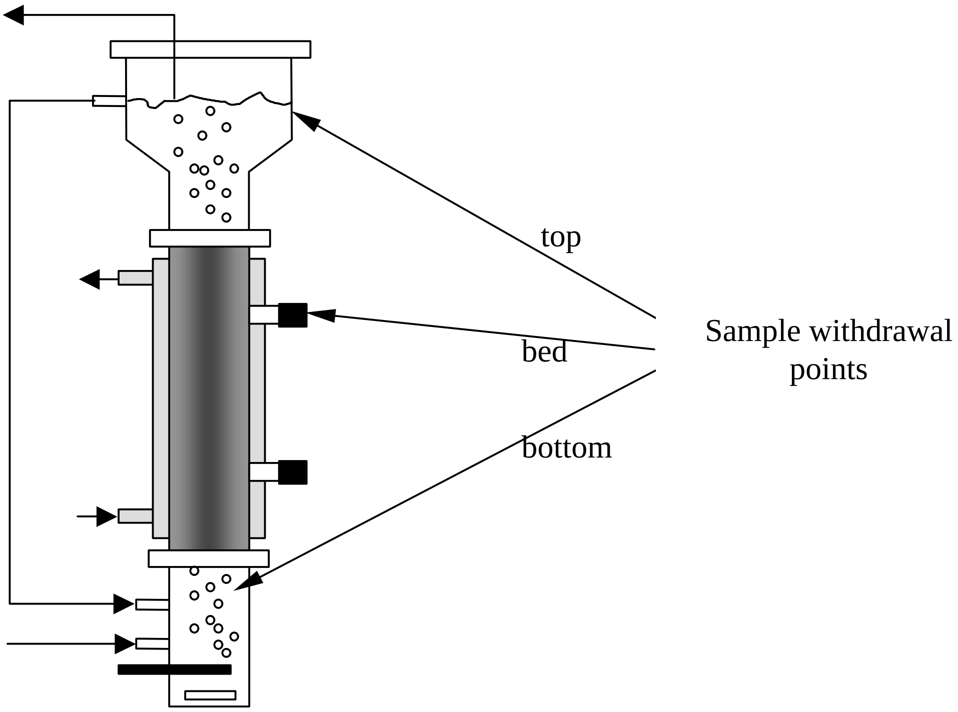


Figure 3.- Points of sampling in the bench packed-bed

Time (min)	Sulfite concentration, bottom section (g/L)	Sulfite concentration, bed section (g/L)	Sulfite concentration, top section (g/L)
2.67		0.399	
5.83	0.404		
10.03			0.396
52.70		0.301	
56.83	0.363		
100.25		0.271	
105.75	0.321		
108.67			0.298
153.42		0.238	
156.30	0.278		
159.98			0.253
204.07			0.221
207.81		0.188	

Table 2.- Experimental evolution of sulfite concentration in the three different sections of the bench column.

1.5.- Numerical calculations to determine the K_La coefficient in each section of the bench column

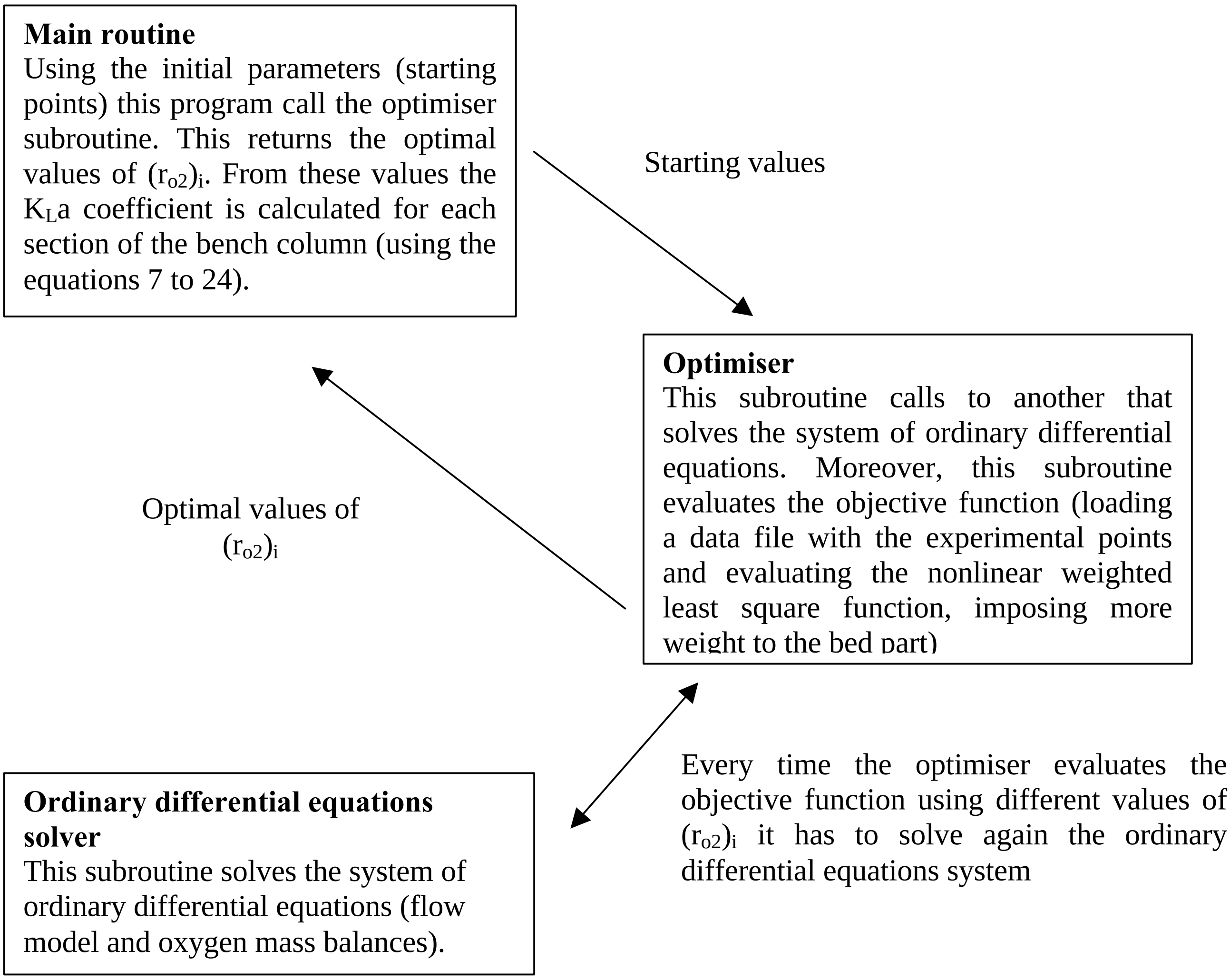
From the mass balances detailed previously (equations 4, 5 and 6) the evolution of the sulfite concentration in the three different sections of the reactor can be estimated if the values of $(r_{o2})_i$ are known. In this way, it is possible to build an objective function to minimise the sum of squares of the differences between the experimental values and the theoretical ones (obtained from the flow model, solving the system of ordinary differential equations). This method is named least square method, and in this case it is employed with a nonlinear model. In addition, weighting factors are used in order to enhance data fitting in the bed section (bioreaction section). The optimisation method is obviously a multivariable method (three parameters to optimise), and also constrained, to avoid results without physical sense. Concretely, the optimisation method used is named ‘constr’ (Matlab v.4.2.).

The importance of good starting values

Good starting values will often allow an iterative technique to converge to a solution much faster than would otherwise be possible. Also, if multiple minima exist or if there are several local minima in addition to an absolute minimum, poor starting values may result in convergence to an unwanted stationary point of the sum of squares surface (Draper, N.R. and Smith, H., 1998).

In this study, the selection of the initial values to start the optimisation method has been made from the expected values of K_La using simple empirical correlations (TN 43.41).

A simple diagram to explain the calculations carried-out to estimate the obtained values after optimisation is detailed in **figure 4**. All computation has been performed using Matlab (v.4.2.).



Main routine
Using the initial parameters (starting points) this program call the optimiser subroutine. This returns the optimal values of $(r_{o2})_i$. From these values the K_La coefficient is calculated for each section of the bench column (using the equations 7 to 24).

Optimiser
This subroutine calls to another that solves the system of ordinary differential equations. Moreover, this subroutine evaluates the objective function (loading a data file with the experimental points and evaluating the nonlinear weighted least square function, imposing more weight to the bed part)

Ordinary differential equations solver
This subroutine solves the system of ordinary differential equations (flow model and oxygen mass balances).

Every time the optimiser evaluates the objective function using different values of $(r_{o2})_i$ it has to solve again the ordinary differential equations system

Figura 4.- Scheme of the computation sequence using the commercial software Matlab v.4.2.

1.6.- Final results of K_La coefficient using the sulfite method

The final results obtained are presented in **figure 5** and **table 3**.

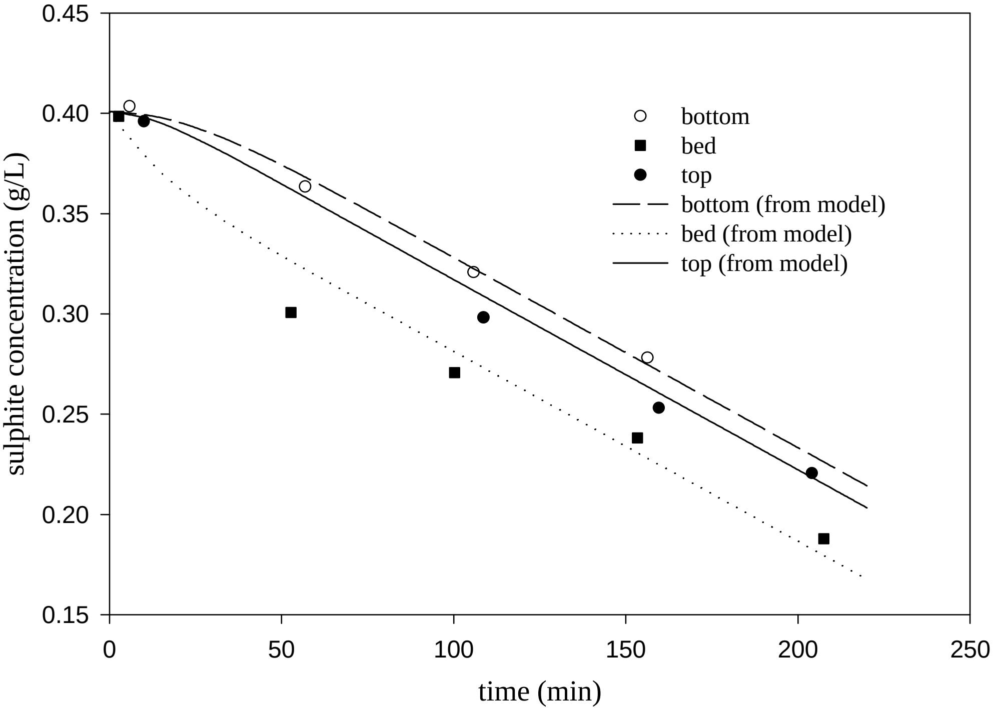


Figure 5.- Results of the fitting curves using nonlinear weighted least squares from the flow model and the experimental evolution of the sulfite concentration in the different sections of the bench column.

reactor section	r_{O2} (mol· L^{-1}· min^{-1})	**K_La (s^{-1})**
bottom	2.0e-5	**1.8e-3**
bed	1.3e-3	**0.19**
top	4.0e-6	**9.3e-4**

Table 3.- Results of the optimisation in the determination of K_La coefficient using the sulfite method

Discussion

Although the fitting curves give a satisfactory concordance to the experimental evolution of the sulfite concentration, taking into account that the K_La coefficient of the bed section has a value much higher than the coefficient values of the other two sections, the precision of the coefficients estimation in the two sections with lower K_La, is notable lesser than in the bed section. This fact occurs due to the mixing effect of the liquid phase. The evolution of the dissolved oxygen concentration (sulfite concentration in the case of the sulfite method) in the top and bottom sections is very influenced by the dissolved oxygen concentration and the K_La coefficient in the bed part (very much higher than in the other two sections).

Conclusions

Through this chemical method, the K_La of the bed has been estimated accurately. On the other hand, the influence of the effect of the liquid phase mixing in the evolution of the sulfite concentration in the bottom and top parts of the bench columns has been explained through the flow model fitted to the residence time distribution experiments.

2.- “GAS-IN GAS-OUT” method using an oxygen dissolved probe

2.1.- Principle

The principle of this method has been described in TN 37.510.

2.2.- Conditions of the experiments and precedents

After the oxygen probe characterisation (**figure 6**, response time 27.5 s), the response time of this probe is used to determine the K_La coefficient by curve fitting of the experimental data to the theoretical oxygen concentration time-profiles, using a non-linear procedure (TN 37.510). The equation used is (Chisti, M.Y., 1989):

$$C_{med} = C^* + \frac{C^* - C_0}{1 - \mathrm{t}\, K_L a}\left[\mathrm{t}\, K_L a \exp\left(-\frac{t}{\mathrm{t}} \right) - \exp\left(-K_L a t \right) \right]$$

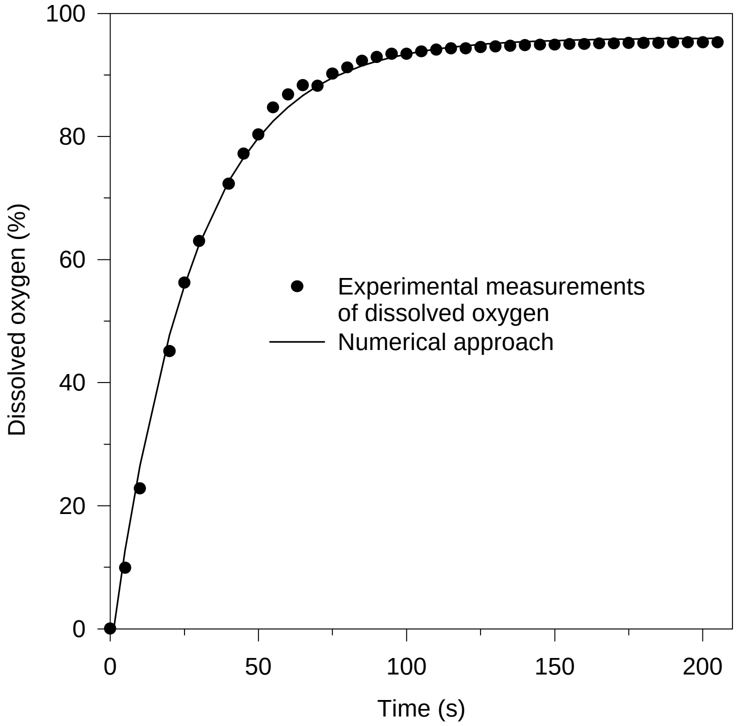


Figure 6.- Characterisation of the oxygen probe, determination of the response time.

In **table 4** the conditions used for the K_La determinations and the summary of the results of the coefficients determined are detailed. The temperature of all determinations is 28 °C.

Air Flow (mL/min)	Stirring rate (r.p.m.)	Rec.Flow (mL/min)	vvm (min^{-1})	K_La (s^{-1})
40	300	4.5	0.08	0.0013

Table 4.- K_La determination with dissolved oxygen probe.

This result does not consider the possible effects of the mixing of the liquid phase. For this reason, the evolution of the oxygen concentration in the liquid phase of each section of the bench column could be affected by the dissolved oxygen concentration of the contiguous sections.

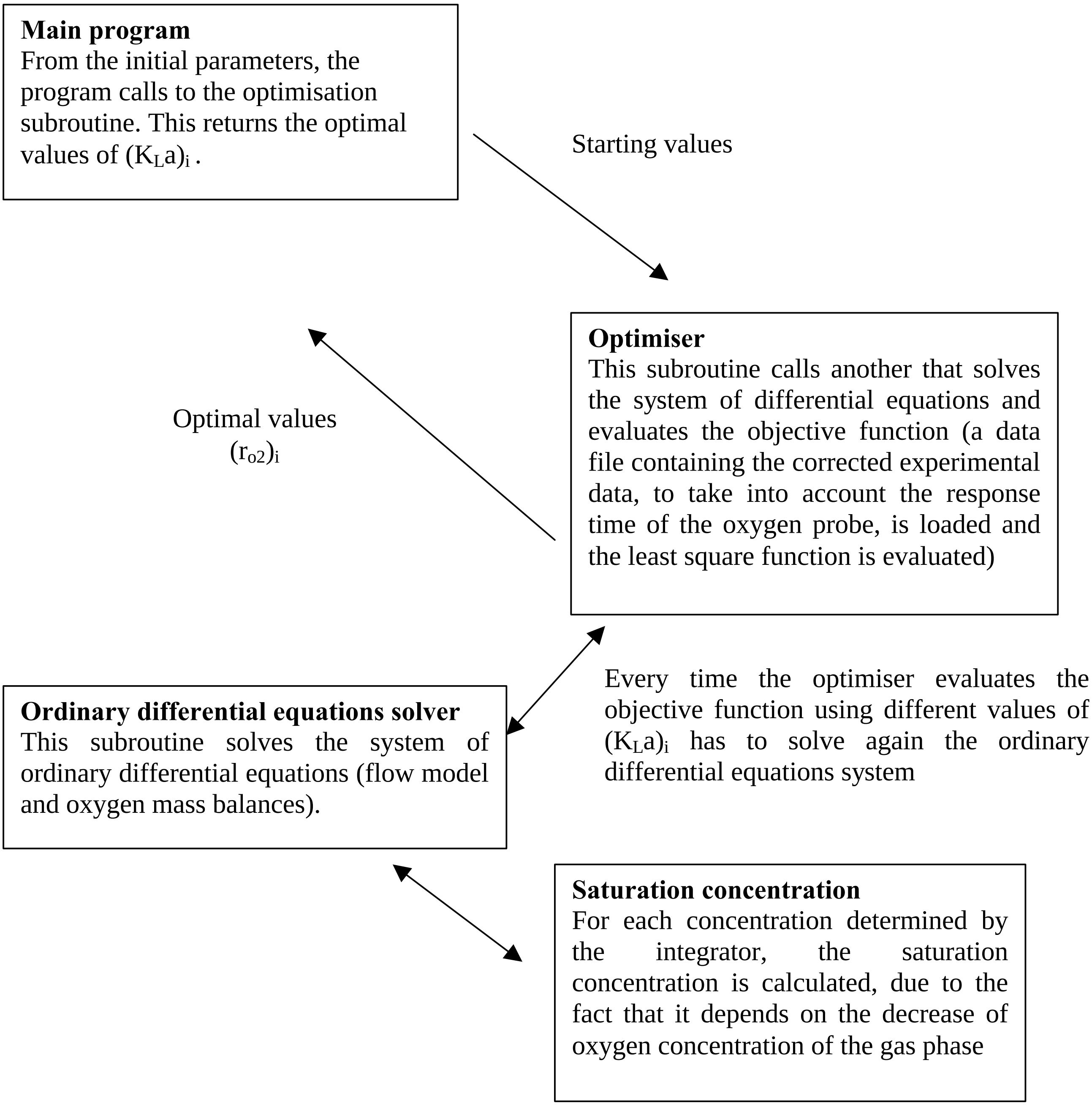
To correct this, like in the case of the sulfite method, it is necessary to introduce the effect of the liquid phase mixing (that is to say, the flow model) in the numerical determination of K_La coefficient.

Taking into account the flow model of the reactor, it is possible to predict the evolution of the oxygen in the top part of the bioreactor, and in this way, the K_La coefficient could be determined, estimating at the same time this coefficient for the two other sections.

Now, the objective function will be the sum of the squares of the differences between the experimental dissolved oxygen concentration (only in the top section) and the theoretical one, estimated from the resolution of the system of ordinary differential equations (flow model). In this case, in the nonlinear least square function it is not possible to introduce the weighting coefficients in the same way that has been performed in the sulfite method for the reason that the experimental data are fully available in the top section of the bench column. Another possibility would be trying to introduce these weighting terms at the experimental data, therefore it would be possible to enhance the fitting of a part of the theoretical results of the model more than the others. As the first results obtained using a non-weighted least squares function showed good concordance with the experimental values, the possibility of introducing weights in the objective function was discarded.

2.3.- Numerical computations and results

The calculations carried-out in this case differ slightly in respect of the sulfite method computation, and a general scheme of the computational sequence used to estimate the K_La in the different sections of the bench column is detailed in **figure 7**. In **figure 8** and **table 5**, the final results of K_La coefficient are presented.



Main program
From the initial parameters, the program calls to the optimisation subroutine. This returns the optimal values of $(K_La)_i$.

Optimiser
This subroutine calls another that solves the system of differential equations and evaluates the objective function (a data file containing the corrected experimental data, to take into account the response time of the oxygen probe, is loaded and the least square function is evaluated)

Ordinary differential equations solver
This subroutine solves the system of ordinary differential equations (flow model and oxygen mass balances).

Saturation concentration
For each concentration determined by the integrator, the saturation concentration is calculated, due to the fact that it depends on the decrease of oxygen concentration of the gas phase

Every time the optimiser evaluates the objective function using different values of $(K_La)_i$ has to solve again the ordinary differential equations system

Figure 7.- Scheme of the computation sequence using the commercial software *Matlab v.4.2*.

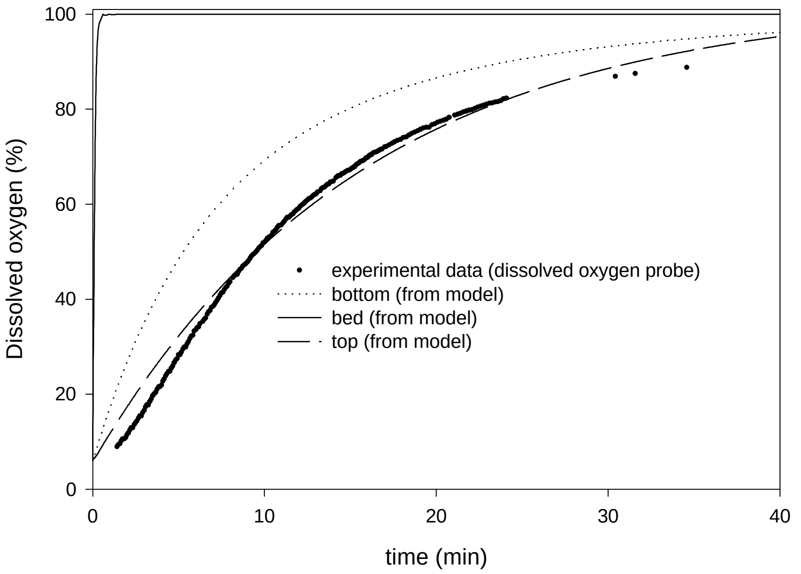


Figure 8.- Results of curve fitting using nonlinear least squares from the differences between flow model and experimental evolution of dissolved oxygen concentration in the top section of the reactor.

Reactor section	**K_La (s^{-1})**
Bottom	**2.1e-3**
bed	**0.20**
top	**6.3e-4**

Table 5.- Results of the optimisation of the K_La coefficient using the method "gas-in gas-out".

Discussion

This method is not accurate to estimate correctly the K_La coefficient in the bed part, because the experimental evolution of dissolved oxygen concentration is only available for the case of the top section of the bench column. On the other hand, the important differences between K_La coefficient along the column cause the increase in the possible error of the values of K_La in the top and bottom parts because the effect of the liquid phase mixing is more important that transference from gas to liquid phase.

Conclusions

This experiment allows to check that the results obtained with the sulfite method can also explain the evolution of the dissolved oxygen concentration in the top part of the column following a different measurement of the oxygen concentration in the liquid phase.

3.- Empirical correlations

In the sulfite method the rate of reaction is assumed zeroth order in Na_2SO_3, but it is not so fast that reaction occurs in the liquid film around the gas bubbles. This would decrease the apparent film thickness and give incorrectly high values of K_La (Blanch and Clark, 1996). For this reason, a brief comparison between experimental results and theoretical calculation of the K_La coefficient has been carried-out.

As an acceptable starting point, the gas phase in the column has a plug flow behaviour in the bed and also in the top parts; only the gas phase in the bottom part could be considered as well-mixed.

For this reason, the starting point to theoretically estimate the K_La coefficient has been the bubble columns. In this way, Blanch and Clark (1996) determined this coefficient in bubble columns using the penetration theory with the following expression:

$$K_La = \sqrt{\frac{4 \cdot D_{O_2} \cdot u_B}{\pi \cdot d_B}}\,\frac{6\phi}{d_B} = \sqrt{\frac{4 \cdot D_{O_2} \cdot u_S}{\pi \cdot d_B \phi}}\,\frac{6\phi}{d_B} = 12\sqrt{\frac{D_{O_2} \cdot \phi \cdot u_S}{\pi \cdot d_B^3}}$$

Therefore, the K_La coefficient depends on the gas holdup (f =*gas volume/liquid volume*), on the superficial gas velocity (u_s) and on the bubble diameter (d_B). The gas holdup was determined experimentally and could be observed that the gas distribution along the column was non-homogeneous (in fact, a high percentage of gas was accumulated in the area just under the bed, see **figure 9**). Due to the reason that the study of the K_La coefficient is necessary to be carried-out separately in the three different sections of the column, the gas holdup was determined also by separate, in these differentiate parts of the bench column (see **table 6**).

Reactor section	Gas holdup (f)	Superficial gas velocity (cm/s)
Top	0.01725	0.02171
Bed	0.05060	0.1278
Bottom	0.4428	0.05878

Table 6.- Gas holdup and superficial gas velocity for each part of the bench column.

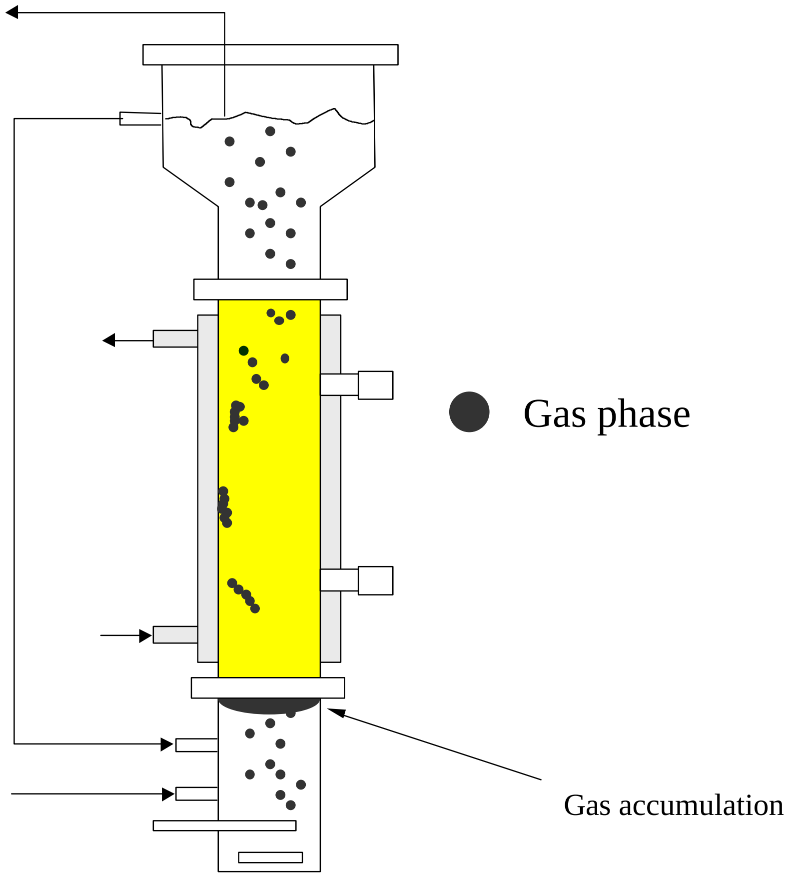


Figure 9.-, Non-homogeneous distribution of the gas phase in the bench column.

Top part

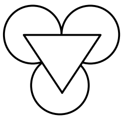
The bubble diameter at the aeration flow-rate studied is approximately between 3-6 mm. With this value of the bubble diameter, the calculated values for the K_La coefficient are:

$d_B = 3$ mm → $\mathbf{K_La = 4 \cdot 10^{-3}\ s^{-1}}$
$d_B = 6$ mm → $\mathbf{K_La = 1 \cdot 10^{-3}\ s^{-1}}$

Bed

The bubble diameter can not be estimated visually like in the top section. For this reason, the maximal bubble diameter has been evaluated using the free space between polystyrene beads and the space between the beads and the column wall.

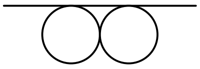
Free space between beads



Making simple geometric calculations it is possible to determine the maximal diameter of the bubbles. Taking an averaged value of the bead diameter (4 mm), this maximal diameter is:

$$\frac{2}{3}\cdot h = r_b - 2\,;\ \ d_B\text{=0.6 mm}$$

In the case of the bubbles that rise between the beads and the glass wall of the column, the maximal bubble diameter is:



$$(2 - r_B)^2 + 2^2 = (2 + r_B)^2$$
$$d_B = 2\cdot r_B = 2\cdot 0.5 = 1\text{mm}$$

Using these values, the corresponding theoretical K_La values are:

d_B = 0.6 mm → **K_La = 0.2 s^{-1}**
d_B = 1.7 mm → **K_La = 0.09 s^{-1}**

Bottom part

This part is a stirred tank, and it is possible to evaluate the K_La coefficient using empirical correlations, but in these common correlations do not appear the magnetic stirrer (the equations are for mechanically stirred reactors). The equation used is:

$$K_L a = 2.6\cdot 10^{-2}\cdot\left(\frac{P_g}{V}\right)^{0.4}\cdot v_S^{0.5}$$

K_La = 0.001 s^{-1}

Discussion

Although the top part of the reactor can be considered a bubble column and the bottom section is a stirred tank (in reference to the gas phase), the packed-bed section has a similar behaviour than a bubble column, but the bubbles are not independent (in that way, figure 9, tries to illustrate this point). For this reason, the equation used above to determine the theoretical value of the K_La coefficient in the bed is not completely suitable. The difficulty of this problem is to find a correlation to determine this transfer coefficient in a fixed bed, with this type of gas flow.

Conclusions

A brief review of the theoretical equations and empirical correlations to estimate the K_La coefficient was essential to try to justify the important differences between the K_La values in the different sections of the bench columns, that had been obtained after the computation of the experimental results with the adaptation of the sulfite method to a reactor with non-ideal flow.

As a summary, all the results obtained after this study are detailed in **table 7**.

Column section	**K_La, sulfite method (s^{-1})**	**K_La, "gas-in gas-out" (s^{-1})**	**K_La, correlations and theoretical calculations (s^{-1})**
Bottom	1.8e-3	2.1e-3	$1\cdot 10^{-3}$
Bed	0.19	0.20	[0.09,0.2]
Top	9.3e-4	6.3e-4	$[1\cdot 10^{-3}, 4\cdot 10^{-3}]$

Table 7.- Summary of results of K_La studies.

4. General conclusions

The methodology defined in this technical note allow to estimate the K_La coefficient in the different sections of the fixed bed reactors used in the third compartment. After discussion of the results obtained with LGCB, new experiments with a more exhaustive number of points must be carried out to have an accurate estimation of the K_La coefficient along the reactor, in addition, it is necessary to carry out a study to determine the precision of the estimation.

On the other hand, to improve the methodology used with the sulfite method a set of considerations will be introduced in the calculations:

- The effect of pressure on the different sections of the bench column.
- The oxygen solubility has to be estimated taking into account the dissolved solutes in the solution used to perform the experiments.
- New experiments have to be performed in continuous mode to assure the correct utilization of the flow model (RTD experiments).

5. References

BLANCH, H.W. and CLARK, D.S.; 1996. Biochemical Engineering. Marcel Dekker, Inc. New York, USA.

CHISTI, M.Y.; 1989. Air-lift bioreactors. Elsevier, New York, USA.

DRAPER, N.R. and SMITH, H.; 1998. Applied Regression Analysis. Ed. Wiley-Interscience. USA.

PÉREZ, J.; MONTESINOS, J.L. and GÒDIA, F. 1998. TN 37.510. Operation of the bench nitrifying reactors.