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Test with bench scale packed-bed reactors

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1. Introduction

The compartment III of the MELISSA loop (nitrifying compartment) consists of a packed-bed reactor with cells of two bacteria strains (*Nitrosomonas europaea* and *Nitrobacter winogradskyi*) immobilised on polystyrene beads (Biostyr). The objective of this compartment is to transform the ammonium ions present in the exit stream from compartment II (*Rhodospirilum rubrum*) into nitrate, a nitrogen source better assimilated by the cells cultured in compartment IV (*Spirulina platensis*).

As discussed previously (WP 25.6), it was decided to set-up three bench scale reactors, in addition to the pilot scale packed reactor, in order to generate more results to fully characterise this system. The detailed design, sterilisation procedure and start-up of these columns were already presented in TN 37.510.

After a long period of operation with two of the bench scale packed-bed reactors, different conditions of ammonium input load, and air flow-rate were tested and discussed in TN 37.520.

The third column was used to perform a detailed physical characterisation of the bench scale nitrifying reactors, and once these studies were finished (new version of TN 37.510), the column was inoculated and its continuous operation started. This reactor will be used as nitrifying compartment in the preliminary connection between the three compartments of the MELISSA loop (WP 43.8, in progress).

A number of environmental factors influence the nitrification, among others, the substrate concentration, temperature, oxygen, pH and possible toxic substances. The main objectives for this workpackage were to investigate the effects of ammonium load in terms of concentration and flow-rate on the ammonium conversion. In order to study and describe quantitatively these effects, NH_4^+ , NO_2^- and NO_3^- concentrations were measured periodically. Among other variables studied, the aeration flow-rate was also studied, allowing to obtain different steady states when increasing or decreasing the aeration. The results obtained during this period are discussed in the present report.

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2. Operational conditions of the experiments

The first phase of the operation of the bench columns, in which the cell attachment and the biofilm formation take place, allows obtaining a system where cells are kept retained inside the reactor. In that way, nitrification process is possible at important dilution rate values, without problems of washing out of the biomass inside the reactor. This initial phase, and the following first steady states attained in the bench nitrifying reactors were described in TN 37.510 and 37.520.

The common conditions of the operation of the two bench columns for all the tests are specified in **table 1** and the variables studied in the experiments are described in the next point.

| VARIABLE | VALUE | | |
|--------------------------------|------------|--|--|
| Temperature | 30 °C | | |
| pН | 8.1 | | |
| Recirculation flow-rate | 8 ml/min | | |
| Stirring | 300 r.p.m. | | |
| Light conditions | darkness | | |

 Table 1.- Culture conditions of the bench columns.

As it has been described in previous technical notes (TN 37.510), the pH control used in these bench reactors adds acid or base to maintain the pH set point; the base used is a solution of sodium carbonate with a concentration of 40 g/L (saturated solution).

The two variables studied in the experiments are :

• The ammonium input load, which was increased until a conversion defining the limitation in the metabolization of ammonium into nitrate. This limitation could be due to the amount of bacteria in the biofilm (in that respect the steady states attained may be really pseudo-stationary states), or due to a poor aeration. It should be said that aeration is usually a limiting step for this conversion. • The aeration flow-rate. An insufficient aeration in a nitrifying reactor is normally easy to identify as an increase of the in the nitrite concentration, that is to say, as a partial nitrification (Garrido, 1996).

3. Experimental results

The bench column number 1 (notation used in the last technical note, TN 37.520), after the last steady state, started to decrease its conversion, and a generalised phenomena of biofilm detachment occurred. The analysis of possible causes for this problem pointed out instability in the pH control, that could not be restored. As a consequence, the operation of column 1 was discontinued, and it was used to perform DTR and K_La experiments in conditions with biofilm on the beads. It should be mentioned that this physical characterisation of the columns had been done previously with the reactors without cell biofilm.

Thus, the experiments that are reported in this technical note were carried out with the bench column number 2 at different operational conditions, basically ammonium loads and aeration flow-rate, as summarised in **table 2**. The evolution of the concentrations of NH_4^+ , NO_2^- , NO_3^- , at different conditions of dilution rate and aeration flow-rate are detailed in **table 3**, figure 1.

| Column | Input medium concentration (g N-NH ₄ ⁺ / L) | Dilution rate (h ⁻¹) | Aeration flow- rate (mL / min) | Ammonium load (g / (L·h)) |
|--------|---|--|--------------------------------------|---------------------------------|
| 2 | 0.300 | 0.10 | 40 | 0.030 |
| 2 | 0.300 | 0.20 | 40 | 0.060 |
| 2 | 0.300 | 0.10 | 100 | 0.030 |
| 2 | 0.300 | 0.20 | 100 | 0.060 |

Table 2.- Operating conditions of the bench column number 2. The aeration in all the cases is with air.

The conversion to nitrate attained using a residence time of 10 h (D= 0.10 h⁻¹) and an aeration flow-rate of 40 mL/min, as it can be observed in figure 1, is very high (approximately 99.7 %). This fact indicates that the oxygen mass transfer from the gas

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to the liquid phase is high enough to maintain a low concentration of nitrite due to its rather complete conversion to nitrate.





Column 2; Step in the dilution rate

Figure 1.- Step in the dilution rate, aeration flow-rate 40 mL/min of air.

When the residence time of the culture operation was decreased to 5 h (D = 0.20 h⁻¹), the conversion to nitrate also decreased to an averaged value of 67.1 %. The concentration of ammonium in the output flow became notable (an averaged value of 0.052 g N-NH₄⁺ / L), and moreover, the nitrite concentration also increased to an averaged value of 0.047 g N-NO₂⁻ / L. The operation of the reactor in these conditions was prolonged for more than 180 residence times. This operation was maintained during this long period of time (38 days) to observe the stability of the operation of the

column, and to know whether the cell biofilm was in a really steady state or not. That is to say, the operation of the column was maintained in these conditions, to assure that a possible augmentation in the amount of cells (a higher development of the biofilm) would not increase the conversion of the bench column.

| Number of residence times | Dilution rate h ⁻¹ | $g N-NH_4^+/L$ | $g N-NO_2^-/L$ | $g N-NO_3^- / L$ | g total N / L | N-balance% |
|---------------------------|----------------------------------|----------------|----------------|------------------|---------------|------------|
| 0 | 0.10 | 0.001 | 0.0003 | 0.302 | 0.303 | 101 |
| 0.68 | 0.10 | 0.001 | 0.0 | 0.303 | 0.304 | 101 |
| 2.75 | 0.10 | 0.001 | 0.0002 | 0.295 | 0.296 | 99 |
| 4.65 | 0.10 | 0.001 | 0.0001 | 0.292 | 0.293 | 98 |
| 14.25 | 0.10 | 0.0 | 0.0007 | 0.307 | 0.308 | 103 |
| 18:89 | 0.20 | 0.040 | 0.058 | | 0.287 | 96 |
| 24.63 | 0.20 | 0.048 | 0.053 | 0.184 | 0.285 | 95 |
| 28.58 | 0.20 | 0.049 | 0.038 | 0.195 | 0.282 | 94 |
| 47.75 | 0.20 | 0.045 | 0.031 | 0.212 | 0.288 | 96 |
| 86.15 | 0.20 | 0.075 | 0.038 | 0.184 | 0.297 | 99 |
| 100.55 | 0.20 | 0.058 | 0.052 | 0.188 | 0.298 | 99 |
| 158.15 | 0.20 | 0.045 | 0.058 | 0.198 | 0.301 | 100 |
| 186.95 | 0.20 | 0.053 | 0.048 | 0.202 | 0.303 | 101 |
| 201,35 | 0.10 | 0,0 | Ó.CODS | 0,229 | 0.220 | 11044×1 |
| 215.35 | 0.10 | 0.001 | 0.0007 | 0.316 | 0.317 | 106 |
| 232.37 | 0.10 | 0.001 | 0.0009 | 0.305 | 0.307 | 102 |

Table 3.- Evolution of the ammonium, nitrite and nitrate concentrations in the output flow of the benchcolumn number 2 for two consecutive changes in the dilution rate, at an aeration of 40 mL/min of air.The first values after every change in the dilution rate are indicated in the table in shadowed cells.

The values of nitrite concentration represent a process of partial nitrification, and this situation is produced due to an insufficient oxygen concentration in the liquid phase (Garrido, 1996). For this reason the first step in order to decrease the nitrite concentration of the output flow was to increase the oxygen concentration by consequently increasing the aeration flow-rate.

First, the conditions of the column were reset to the initial conditions (a residence time of 10 h) to check if the last conditions had altered the viability of the bacteria. After more than 30 residence times, the conversion was maintained in a high value (an averaged value of 99.7 %) very similar to the results obtained for the same

conditions at the beginning of this test. Thus, the repeatability of the steady states reached previously were demonstrated as well as the robustness of the nitrification system.

The following experiment was carried-out increasing the aeration flow-rate up to 100 mL/min, trying to avoid a limitation of oxygen transfer in the degradation process of the ammonium into nitrate. The operating conditions for the continuous culture are detailed in table 2, and the results of the analytical determinations of ammonium, nitrite and nitrate concentrations of the performed experiment are presented in **table 4** and in **figure 2**.

| Number of residence times | Dilution rate h ⁻¹ | g N-NH ₄ ⁺ / L | $g N-NO_2^-/L$ | $g N-NO_3^- / L$ | gtotal N / L | N-balance% |
|---------------------------|----------------------------------|--------------------------------------|----------------|------------------|--------------|----------------|
| 0 | 0.10 | 0.004 | 0.0008 | 0.290 | 0.295 | 99 |
| 14.40 | 0.10 | 0.001 | 0.0 | 0.311 | 0.312 | 104 |
| 28.80 | 0.10 | 0.001 | 0.0001 | 0.300 | 0.301 | 100 |
| 45.60 | 0.10 | 0.001 | 0.0001 | 0.297 | 0.298 | 99 |
| 52.80 | 0.10 | 0.001 | 0.0001 | 0.287 | 0.288 | 96 |
| 93.60 | 0.10 | 0.001 | 0.0001 | 0.285 | 0.286 | 95 |
| 100.8 | 0.10 | 0.0001 | 0.001 | 0.297 | 0.298 | 99 |
| 101.47 | 0.20 | 0.020 | 产生 0.027 4 2 4 | 0.228 | 0.275 | *** 192 |
| 102.15 | 0.20 | 0.048 | 0.055 | 0.183 | 0.286 | 95 |
| 103.20 | 0.20 | 0.061 | 0.068 | 0.150 | 0.279 | 93 |
| 105.62 | 0.20 | 0.062 | 0.057 | 0.169 | 0.288 | 96 |
| 106.92 | 0.20 | 0.064 | 0.044 | 0.187 | 0.295 | 98 |
| 112.53 | 0.20 | 0.058 | 0.024 | 0.201 | 0.283 | 94 |
| 120.00 | 0.20 | 0.067 | 0.016 | 0.205 | 0.287 | 96 |
| 122.45 | 0.20 | 0.079 | 0.016 | 0.200 | 0.295 | 98 |
| 127.00 | 0.20 | 0.080 | 0.017 | 0.190 | 0.287 | 96 |
| 131.92 | 0.20 | 0.074 | 0.018 | 0.194 | 0.285 | 95 |
| 137.13 | 0.20 | 0.067 | 0.016 | 0.197 | 0.279 | 93 |
| 151.57 | 0.20 | 0.070 | 0.014 | 0.212 | 0.296 | 99 |
| 155.93 | 0.20 | 0.065 | 0.014 | 0.212 | 0.291 | 97 |

 Table 4.- Evolution of the ammonium, nitrite and nitrate concentrations in the output flow of the bench column number 2 for two dilution rates at an aeration of 100 mL/min of air. The first values after the change in the dilution rate are indicated in the table in shadowed cells.

In figure 2, the results for the two steady states attained with the bench column number two, working with an aeration flow-rate of 100 mL/min are presented. The conversion obtained when the bench column operates with a residence time of 10 h (D = 0.10 h^{-1}) is very similar to the obtained when the column had an aeration flow-rate of 40 mL/min (99.7 %). Using a step change in the dilution rate, the residence time of operation was decreased to 5 h, then a transient period take place, and after 24 residence times (5 days), the ammonium, nitrite and nitrate concentrations remained rather constant, and therefore, were considered as steady-state values. The averaged ammonium concentration in the output flow is very similar to the one attained in the first experiment: 0.068 gN-NH₄⁺/L. However, the nitrite concentration was quite different, being its average value 0.016 g N-NO₂⁻/L, remarkably smaller than the value obtained for 40 mL/min aeration, about 0.050 g N-NO₂⁻/L.



Column 2; Step in the dilution rate Aeration flow-rate: 100 mL/min

Figure 2.- Step in the dilution rate (0.10 h⁻¹, 0.20 h⁻¹). Aeration flow-rate 100 mL/min of air.

Therefore, the residual nitrite concentration had decreased when the aeration flow-rate had been increased, although this concentration under these conditions still showed that oxygen mass transfer could still be the limiting factor for the nitrification process. However, these results are valuable for confirming that the higher the aeration flow-rate is used, the better the conversion into nitrate is obtained.

For this reason, the next experiment planned to be performed using this nitrifying bench reactor will operate with a higher aeration flow-rate, to avoid the oxygen mass transfer being limiting for the nitrifying process.

On the other hand, the averaged value of conversion to nitrate during this second experiment is 70.8 %; this value has been slightly increased comparing with the first steady state attained (e.g. aeration of 40 mL/min, 67.1 % of conversion at 5 h of residence time).

A summary of the results obtained with the nitrifying bench column in these two long-term experiments described is presented in **table 5**. Averaged values of conversion into nitrate (removal efficiency) and of partial conversion to nitrite (remained $N-NO_2^{-1}$) in the different steady states attained are detailed in this table.

| Experiment number | Aeration flow-rate | Residence time (h) | Input conc. g/L N-NH₄⁺ | Removal efficiency to nitrate (%) | Remained N-NO ₂ ⁻ (%) |
|----------------------|-----------------------|-----------------------|---------------------------|---|---|
| 1 | 40 mL/min | 10 | 300 | 99.7 | 0.3 |
| | 40 mL/min | 5 | 300 | 67.1 | 15.7 |
| 2 | 100 mL/min | 10 | 300 | 99.7 | 0.3 |
| | 100 mL/min | 5 | 300 | 70.8 | 5.3 |

 Table 5.- Summary of results of conversion obtained in the different experiments carried out with the bench column number 2.

The variation of the conversion to nitrate obtained in the two experiments is presented in **figure 3**. It is clear that at a residence time of 10 h, the conversion is very high. However, when the bench column operates at a residence time of 5 h the



conversion obtained is lower, due to a possible, at least partially, oxygen mass transfer limitation.

Figure 3.- Evolution of the conversion (to nitrate and partial to nitrite). Bench column 2.

4. Comparison between nitrifying reactors

The experiments previously reported with the bench column number 2 are performed in the same conditions of ammonium load (0.060 g N-NH₄⁺/L h) that an experiment conducted at the Pilot Nitrifying Reactor (reported in TN 37.420). The results obtained in the experiment of the pilot column (**figure 4**), can be compared with the bench column results (figures 1 and 2), especially taking into account the differences between the aeration conditions used in each reactor. For this reason, in **table 6** and **figure 5** the conversions to nitrate and the partial conversion to nitrite attained in both reactors during the three different experiments are detailed. While the Pilot Reactor of the third compartment of the Melissa loop achieves almost total degradation of the input ammonium load to nitrate (only for a transient period after the step change in the residence time, a certain accumulation of NO2- due to partial conversion of the ammonium could be observed), the bench column do not achieve it neither for an aeration of 40 mL/min nor for an of 100 mL/min.

Nitrifiying pilot reactor



Figure 4.- Evolution of the ammonium, nitrite and nitrate concentrations in a step of the residence time (from 10 to 5 hours) in the Pilot Reactor.

| Reactor | Aeration v.v.m. (air / liquid) | Residence time (h) | Input conc. g/L N-NH₄ ⁺ | Removal efficiency to nitrate (%) | Residual N-NO2 ⁻ (%) |
|---------|--------------------------------------|-----------------------|---------------------------------------|--|---------------------------------------|
| Pilot | Automatic control | 10 | 0.300 | 99.7 | 0.3 |
| column | (pure oxygen) ; 0.8 vvm | 5 | 0.300 | 99.7 | 0.3 |
| Bench | Constant input flow-rate | 10 | 0.300 | 99.7 | 0.3 |
| column | (air) ; 0.08 vvm | 5 | 0.300 | 67.1 | 15.7 |
| Bench | Constant input flow-rate | 10 | 0.300 | 99.7 | 0.3 |
| column | (air) ; 0.2 vvm | 5 | 0.300 | 70.8 | 5.3 |

 Table 6.- Comparison between nitrifying reactors. Differences between conversions operating at a same input ammonium load but using different aeration conditions.

It must be pointed out the fact that although the residual N-NO₂ % decrease when increasing the vvm, the conversion to nitrate do not increase in a large extent as can be expected in a reaction-in-series system ($NH_4^+ -> NO_2^- -> NO_3^-$). As a result, the ammonium conversion calculated as the sum of the conversion to nitrate and partial conversion to nitrite, decreased, showing the complex interactions between reactions involved and their limiting factors.

Among other possible explanations for this fact, there is the different closure of the nitrogen balance for the two experiments. In the 0.08 vvm experiment the N-balance is closed rather completely, while in the 0.2 vvm only 96 % of closure is achieved. The different closure of the N-balance was already discussed as well as its possible relations with denitrification processes involved in oxygen limited cultures of *Nitrosomonas* and *Nitrobacter* (TN 37.420 and TN 37.520). If the overall ammonium conversion is now calculated in terms of remaining ammonium with respect to the initial ammonium in the input flow, the differences become minimal.



Figure 5.- Conversion obtained with the different nitrifying reactors, at the same conditions of input ammonium load and residence time of 5 h.

5. Experimental determination of K_L coefficient in a bench column with mature biofilm

Once the impossibility of the nitrifying normal operation in column 1 occurred (as it has been explained in page 6), experiments of physical characterisation of the column with biofilm onto the support surface were carried out in this column.

Determinations of the K_La coefficient were performed to verify if differences between the K_La coefficient without and with biomass onto the support surface happened. The method used to make these determinations is the well-known 'dynamic method' (van't Riet, 1979). In this method the aeration is switched off to measure the decrease of the dissolved oxygen concentration (this decrease is due to the oxygen consumption by the biomass). Then, once the dissolved oxygen concentration in the liquid phase has decreased significantly, the aeration is switched on again, and the increase of the dissolved oxygen concentration is measured. In this way, the determination of the oxygen consumption and the K_La coefficient is possible.

The bench column was maintained in continuous operation (with a very low dilution rate), and the conditions used in the experiment of determination of the K_La coefficient are detailed in **table 7**. With the low dilution rate used in the bench column the oxygen consumption rate was very low, and once the aeration was switched off, the dissolved oxygen concentration was kept quite constant during more than 15 minutes. Therefore, the oxygen consumption at this dilution rate (0.02 h⁻¹) can be neglected. The method used to determine the K_La coefficient is the same described in TN 25.330 and TN 37.510. The oxygen present in the liquid phase is purged with a nitrogen flow, and once the oxygen concentration is low enough, the aeration is returned to his old value. From the evolution of the oxygen concentration and the response time of the oxygen probe (τ) is possible to determine the K_La coefficient, from the equation:

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

| VARIABLES | VALUES |
|-------------------------|----------------------|
| Aeration flow-rate | 40 - 250 mL/min |
| Recirculation flow-rate | 4.5 mL/min |
| Stirring | 300 r.p.m. |
| Dilution rate | 0.02 h ⁻¹ |
| Temperature | 28 - 24 °C |
| pH | 8.1 |

Table 7.- Operating conditions of the experiments of the K_La coefficient determination in the bench column with mature biofilm.

One of the experiments is presented in **figure 6**, and the coefficient values obtained are presented in **table 8**. Moreover, this table presents the results of the determination of the K_L a coefficient in the same conditions, operating with a column without bacteria attached onto the support surface.



Figure 6.- Experimental determination of the K_La coefficient.

| Aeration | Temperature | K _L a | K _L a |
|-----------|-------------|-------------------|------------------------|
| flow-rate | (°C) | (without biofilm) | (mature biofilm) |
| (mL/min) | | s ⁻¹ | S ⁻¹ |
| 40 | 28 | 0.0013 | 0.0019 |
| 40 | 24 | Not determined | 0.0016 |
| 250 | 28 | 0.0072 | 0.0054 |

 Table 8.- K_La coefficient values at different conditions of aeration flow-rate and temperature. Comparing bench reactors with and without biofilm onto the support surface.

Once the results of the K_La determinations are analysed, two important influences in the values of this coefficient can be observed. In the main body of the bench column, where the biofilm is proliferating, a higher superficial velocity of the gas phase makes possible that important differences of the K_La along the different sections of the reactor appear. The lower expected value of the K_La coefficient is in the top section of the column, where the influence of the stirring is almost negligible and the cross-section area of this zone is really very much bigger than for the other reactor parts.

On the other hand, the influence of the operation temperature in the value of the coefficient allow to affirm that the real oxygen transfer overall coefficient may be higher than the determined. It is important to note that the temperature of the column is kept constant in the area of the jacket, and that the oxygen probe is placed in the top section (where the K_La values are determined). In the top section the temperature is always slightly lower than in the section where the temperature control is working, that is the central part of the reactor, holding solid support beads.

Taking into account the discrepancies observed between the values of the K_La determined, and the oxygen requirements for the rather complete ammonium conversion showed (Poughon, 1998), it is possible to affirm that K_La coefficients are only qualitatively representative of the real values for the zone where the biological degradation is taking place. Anyway, they can be useful in order to detect possible oxygen limitation problems as well as to define how aeration flow-rate, temperature and other physical parameters can affect oxygen mass transfer.

When the maximum theoretical values for oxygen supply in the packed-bed, calculated from the K_La values measured previously, were compared to the actual oxygen consumption based on the reactor performance and the stoichiometry of the reaction, a clear discrepancy appeared as discussed with Clermont-Ferrand group (L. Poughon, 1998). The analysis of the problem suggests that quite probably the K_La values estimated previously are not representative of the K_La in the packed-bed zone of the reactor. This would be due basically to the different superficial velocities of the gas in the packed-bed area, and the top section, where the measurement was done.

For unstirred fermenters (for example: bubble column fermenter) one published correlation for the K_La coefficient (Atkinson and Mavituna, 1983) is:

$$\begin{split} \mathbf{K}_{L} \mathbf{a} &= \alpha \, \mathbf{V}_{g}^{\beta} \qquad \qquad \textit{equation 1} \\ \\ \frac{\mathbf{K}_{L}^{*} \mathbf{a}}{\mathbf{K}_{L} \mathbf{a}} &= \frac{\mathbf{V}_{g-\text{fixbed}}}{\mathbf{V}_{g-\text{toppart}}} = \gamma \end{split}$$

Where \underline{V}_g is the superficial gas velocity expressed in terms of metres per second. The values of $\underline{\beta}$ are around of the unity, whereas the values of $\underline{\alpha}$ depend on the nature of the additives (in our case, this parameter will have relationship mainly with the medium composition and the free cell concentration).

In **table 9**, a series of data is presented for the K_La values. In this table, the real values determined experimentally in the top section (K_La) are compared to those corrected (K_L^*a) according to equation 1 and the V_g calculated for the two sections of the reactor, taking into account geometrical factors and bed porosity.

| Aeration flow- rate (mL/min) | Bed porosity | V _g (packed- bed part) (cm/s) | V _g (top part) (cm/s) | γ (corrector factor) | K _L a (s ⁻¹) | $\mathbf{K}_{\mathbf{L}}^{*}\mathbf{a}\left(\mathbf{s}^{-1}\right)$ |
|------------------------------------|---------------------|---|-------------------------------------|----------------------------|--|---|
| 9 | 0.49(1) | 1.62 | 0.188 | 8.60 | 0.00046 | 0.0040 |
| 9 | 0.40 ⁽²⁾ | 1.98 | 0.188 | 10.5 | 0.00046 | 0.0049 |
| 15 | 0.49 | 2.70 | 0.314 | 8.60 | 0.00070 | 0.0060 |
| 15 | 0.40 | 3.31 | 0.314 | 10.5 | 0.00070 | 0.0074 |
| 40 | 0.49 | 7.20 | 0.837 | 8.60 | 0.0013 | 0.0112 |
| 40 | 0.40 | 8.82 | 0.837 | 10.5 | 0.0019 | 0.0200 |
| 100 | 0.49 | 18.0 | 2.09 | 8.60 | 0.0032 | 0.0275 |
| 100 | 0.40 | 22.0 | 2.09 | 10.5 | 0.0032 | 0.0336 |
| 250 | 0.49 | 45.0 | 5.23 | 8.60 | 0.0072 | 0.0619 |
| 250 | 0.40 | 55.1 | 5.23 | 10.5 | 0.0054 | 0.0567 |

Table 1.- Superficial gas velocity calculations to correct the K_La coefficient (K^*_La). ⁽¹⁾ Bed porosity without biofilm, Forler, 1994; ⁽²⁾ bed porosity with mature biofilm; approximated value, considering common porosity values.

The experimentally determined K_La values correspond to the top part of the bench column, where the diameter is 78 mm (while the diameter of the other column parts is 38 mm). The superficial gas velocity in the top part is quite different of the packed-bed part, mainly due to the effective section (the section of the packed-bed part has to be corrected by the bed porosity, including the biofilm thickness) and of course due to the mentioned differences between diameters. This different superficial gas velocity means that the K_La values are also quite different. In fact, taking $\beta = 1$, the K_La coefficient is directly proportional to the superficial gas velocity. Thus, the equations used to make the calculations of table 1 are:

 $V_{g} = \frac{Q_{aeration}}{S_{effective}} \qquad S_{effective} = SE \qquad S = \frac{\pi}{4}D^{2} \qquad E \cong 0.40 - 0.49$ $\gamma = \frac{V_{g-fixedbed}}{V_{g-toppart}} \qquad K_{L}^{*}a = \gamma K_{L}a$

Once these calculations have been assumed, in case of an aeration flow-rate of 15 mL/min, 0.3 g N-NH₄⁺/ L (in the input flow) and a flow-rate of liquid of 0.8 mL/min, the calculated oxygen mass transfer is high enough to corroborate the experimental values of conversion into nitrate obtained in the experiments reported in TN 37.520:

• Maximal oxygen transfer (C*= $2.74 \cdot 10^{-4} \text{ mol/L}$): RO_{2 max} = K^{*}_La·C* = (0.0060·3600)h⁻¹ · 2.74·10⁻⁴mol/L = 0.0059 mol/(L·h) 0.0059 mol/(L·h) x 0.472 L = **0.0028** mol oxygen /h

• Input ammonium flow = 0.0010 mol ammonium /h

Then, following the reaction:

 $NH_3 + 2O_2 \rightarrow HNO_3 + H_2O$; then $2O_2$ for $1 NH_3$, thus, the relation is:

2.8 mol O_2 / mol N > 2 mol O_2 / mol N

6. Conclusions and future work

Tests with different levels of dissolved oxygen (vvm), studying the effect for both transient and stationary states were conducted observing how this parameter can affect the ammonium conversion and nitrite and nitrate concentrations. The results show that nitrification with immobilised cells is mainly controlled by mass transfer of oxygen, in particular between gas and liquid phase. If enough oxygen is provided to the liquid phase, nitrification conversion was rather complete for the range of ammonium load studied (until 0.120 kg/(m³ h).

Direct consequences of load changes in continuous mode were appreciated when analyses of nitrogen compounds were made. Sometimes mass balance was not accomplished for the liquid phase, and undesirable levels of reaction intermediates, such as nitrite, appeared in the transient phenomena involved.

Determinations of the K_La coefficient were performed to verify if differences between the K_La coefficient without and with biomass onto the support surface appeared. Differences observed were justified by a higher superficial velocity of the gas phase making possible that important differences of the K_La along the different sections of the reactor appeared.

In addition, taking into account the discrepancies observed between the values of the K_La determined, and the oxygen requirements for the rather complete ammonium conversion it is possible to affirm that K_La coefficients are only qualitatively representative from where the biological degradation is taking place, but being useful to detect possible oxygen limitation problems and how physical parameters affect.

From the results presented in this TN 43.410 additional experiments concerning the improvement of the ammonium conversion by increasing aeration flow-rate are planned. It is interesting to check if using similar v.v.m. than in the pilot reactor the conversion reaches similar values when highest ammonium load is applied. In previous experiments the aeration flow-rate has been increased until 0.2 v.v.m., but the maximum employed in the pilot reactor was 0.8 v.v.m. presenting quite complete conversion. This limit of aeration has to be tested for the bench reactors.

Other aspects to be analysed when obtaining complete ammonium conversion can be the accomplishment of the N-balance, minimum nitrite concentration and maximum ammonium load allowed. Finally a global comparison between bench columns and pilot reactors in terms of ammonium conversion versus ammonium load will be presented and discussed.

During the realisation of the tests proposed the variables considered as key parameters for the system will have to be measured, checked and registered in order to explain the reactors behaviour. These variables include pH, dissolved oxygen, temperature, ammonium concentration, nitrate and nitrite concentrations, and also input/output flow-rates.

With these additional tests planned, it could be possible to confirm the proposed bioreaction system is a good alternative to be implemented as the Compartment III in the MELISSA loop, with high conversion and stability, even for long term operations.

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