Universitat Autònoma de Barcelona
Dep. Enginyeria Química
08193 Bellaterra, Barcelona, Spain

MELISSA

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Test with bench scale packed-bed reactors

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PÉREZ, J.; MONTESINOS, J. L. ; GÒDIA, F.

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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 bacterial 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*), and by higher plants.

As discussed previously, 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.

The first phase of the operation of the bench columns, in which the cell attachment and the biofilm formation takes place, allows to obtain a system where cells are retained inside the reactor. In that way, nitrification process is possible at high 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 the results of the operation at different conditions of ammonium input load, and air flow-rate were tested and reported in TN 37.520.

In a next set of continuous runs in the bench scale packed-bed reactors, the attention was focussed on environmental factors, specifically the aeration rate (critical for this compartment) and the recirculation rate. One group of results has been presented

already in TN 42.410. To complete those results, the experiments presented here have the goal to study the effect of the recirculation flow-rate on the conversion of the bench scale nitrifying reactors in both transient and steady states. Although the RTD experiments already carried out with these reactors (TN 37.510.) demonstrated that the reactors have an important degree of mixing, they show a certain deviation from a mixed tank. The recirculation flow-rate notably affects the liquid phase mixing of the reactor. Consequently, an experiment was performed to study the effect of recirculation on the ammonium, nitrite and nitrate concentrations along vertical axis of the bench scale nitrifying reactor.

It is therefore meaningful to test the effect of the balance between the dynamics of the mixing conditions in the reactor and the rate of the biological reaction, that should be reflected in axial concentration profiles when the process would be controlled by a low mixing, and in almost homogeneous profiles when the degree of mixing is high and the whole process is controlled by the reaction rate. In order to investigate these effects, the concentration profiles in the reactor will be analysed by taking samples through the side sampling ports.

2. Operational conditions of the experiments

The conditions of the operation of the bench column during the experiments are specified in table 1.

As it has been described previously, the pH control used in these bench reactors adds either acid or base to maintain the pH set point; the used base is a solution of sodium carbonate with a concentration of 40 g/L (saturated solution at room temperature).

EXPERIMENTAL CONDITIONS OF BENCH COLUMN C2	
Residence time	10 h
Aeration	75 mL/min
Stirring	300 r.p.m.
Temperature	30 ºC
Recirculation	7 →1.4 mL/min (flow-rate)
	1:10→1:2 (feed/rec. ratio)

Table 1.- Operating conditions used in the study of the effect of the recirculation flow-rate in the nitrogen compounds concentration along the fixed bed.

3. Experimental results

The obtained steady states are presented in table 2. In this table the ammonium input load and the corresponding removed load are detailed per unit of total reactor volume.

Tabla 2.- Steady states attained in the bench column reactor during the study of the effect of the recirculation flow-rate.

Recirculation flow-rate (mL/min)	Reciculation ratio (q_{feed}: q_{rec})	Input ammonium load (kg N· m^{-3}· day^{-1})	Output ammonium load (kg N· m^{-3}· day^{-1})	Output nitrite load (kg N· m^{-3}· day^{-1})	Ammonium removed load (%)	Conversion to NO_3^- (%)
7	1:10	0.55	0.0018	0.0012	99.7	99.5
1.4	1:2	0.55	0.0014	0.0003	99.8	99.7

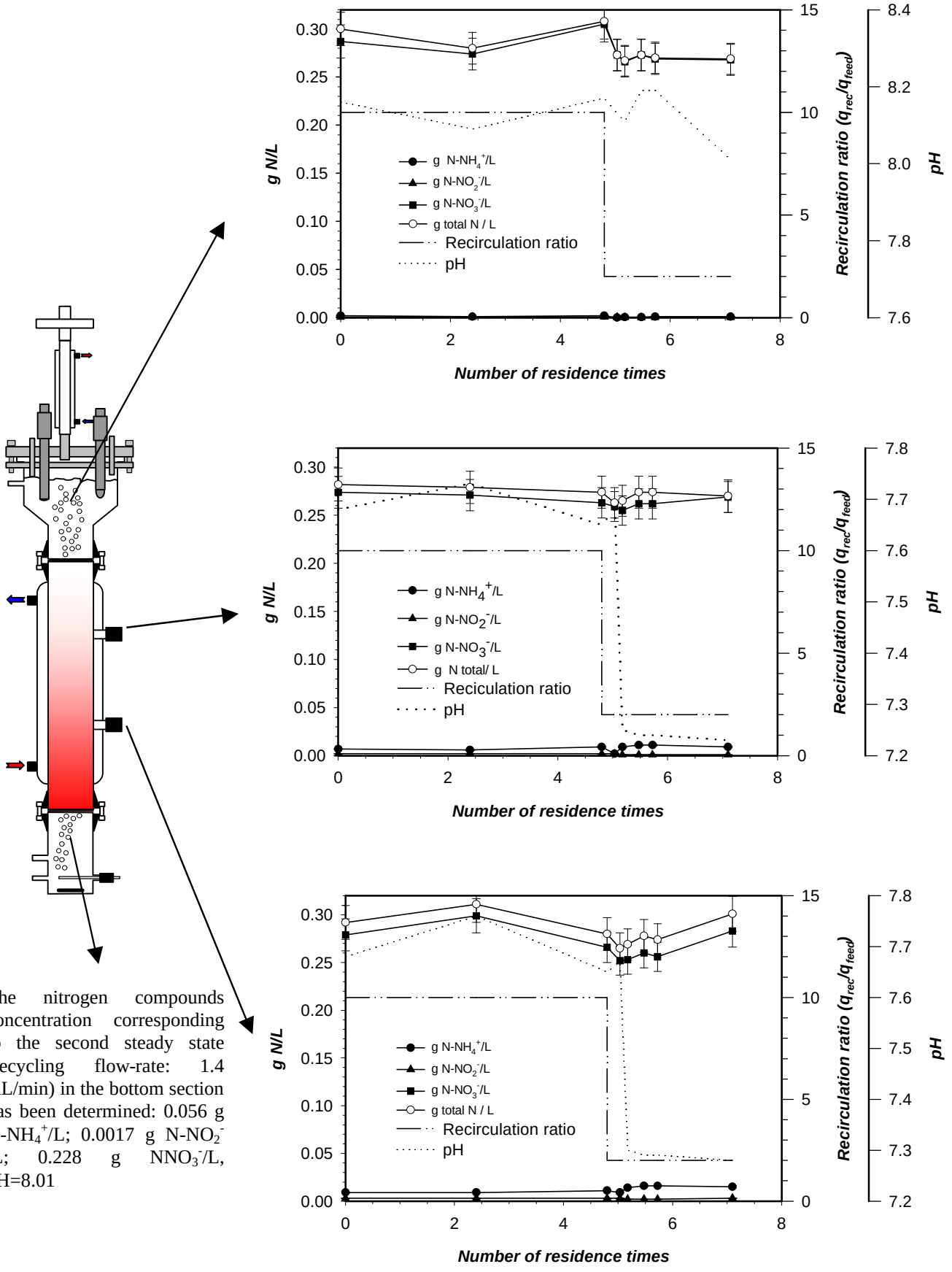
In figure 1 the evolution of the ammonium, nitrite and nitrate concentrations during the recirculation flow-rate step is shown (all the experimental data collected dring these experiments are provided as an appendix at the end of this technical note).

The samples were withdrawn from four different points along the vertical axis of the column:

- Top section, in the same way than in the rest of the experiments reported in technical notes 37.510., 37.520 and 43.41.
- Top lateral port placed in the bed.
- Bottom lateral port, placed in the bed.
- Bottom section of the bench reactor (from this position only a sample was withdrawn, corresponding to the second steady state).

As can be observed in figure 1, no significant variations in the obtained conversion of the bench scale nitrifying reactors are produced during the experiment. The ammonium, nitrite and nitrate concentrations are kept constant along the vertical axis of the bench column, this fact may indicate that the degree of mixing of the liquid phase in the reactor is important, as RTD experiments it had shown.

It is also important to discuss the evolution of pH in this experiment. The pH is controlled in the top section of the reactor (where pH probe is placed, and where the additions of sodium carbonate are performed), but in the bed, the pH are very influenced by the recirculation flow rate used. During the first steady state with recirculation flow-rate of 7 mL/min, the pH value is kept in the range 7.7-8.1 along all the reactor. In contrast, when recirculation flow-rate is reduced at a value of 1.4 mL/min, the pH in the bed decreases to 7.3 in the packed bed, while it is maintained higher at the top. This fact indicates the need to use a high recirculation flow rate in the operation of the nitrifying reactors.



The nitrogen compounds concentration corresponding to the second steady state (recycling flow-rate: 1.4 mL/min) in the bottom section has been determined: 0.056 g $N\text{-}NH_4^+/L$; 0.0017 g $N\text{-}NO_2^-/L$; 0.228 g NNO_3^-/L, pH=8.01

Figure 1.- Step perturbation of the recirculation flow-rate. Effect along the vertical axis of the bench reactor. The residence time used is 10 h.

4. Conclusions

The analysis of the experiment shows that no effects in the overall removal efficiency of the column were detected after the perturbation of the recirculation flow-rate. Moreover, the slight variations in concentration of nitrogen compounds along the column demonstrate that the mixing degree of the liquid phase is important, as RTD experiments had also shown.

On the other hand, the evolution of the pH along the vertical axis of the column during the mentioned perturbation in the recirculation flow-rate, suffers an important variation (decrease) in the bed section mainly due to two reasons. First, these bench scale bioreactors have an only pH probe, placed in the top probe, fact that limits the action of the acid/base control loop. Second, the production of protons due to the degradation of the ammonium by *Nitrosomonas europaea* is located in the bioreaction section (bed). When the recirculation flow-rate decreases, the mixing of the sodium carbonate (base solution, which is added in the top section) in the total volume of the reactor is slower. Although at the ammonium load studied no effects in the removal efficiency of the bioreactor were detected, this pH variation in the bed section could affect conversion for higher ammonium loads, and therefore too low recirculation rates should be avoided in the operation of this reactor.

These reactors clearly reflect the different dynamics of these three processes: mixing, in the reactor acid base generation / addition, and biological reaction. When the recirculation rate is high, mixing time is the lowest characteristic time of the three processes, and both pH and internal profiles are not observed. For a low recirculation rate, the mixing time decreases to an extent that affect pH homogeneity, but does not affect importantly the biological reaction at the fixed ammonium load.

5. References

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Appendix

Time (h)	Number of residence times	Top section			
		g NNH_4^+/L	g NNO_2^-/L	g NNO_3^-/L	g total N/L
0	0	0.002	0.0006	0.287	0.300
24	2.4	0.001	0.0005	0.274	0.280
48	4.8	0.002	0.0008	0.305	0.308
50.33	5.033	0	0.0003	0.273	0.273
51.75	5.175	0.0005	0.0002	0.266	0.267
54.75	5.475	0.0005	0.0001	0.273	0.273
57.25	5.725	0.001	0.0002	0.269	0.270
71	7.1	0.001	0.0002	0.268	0.269

Time (h)	Number of residence times	Top lateral port (bed)			
		g NNH_4^+/L	g NNO_2^-/L	g NNO_3^-/L	g total N/L
0	0	0.007	0.002	0.274	0.282
24	2.4	0.006	0.002	0.271	0.279
48	4.8	0.009	0.002	0.263	0.274
50.33	5.033	0.002	0.002	0.259	0.263
51.75	5.175	0.009	0.001	0.255	0.265
54.75	5.475	0.011	0.001	0.262	0.274
57.25	5.725	0.011	0.001	0.262	0.274
71	7.1	0.009	0.001	0.269	0.27

Time (h)	Number of residence times	Bottom lateral port (bed)			
		g NNH_4^+/L	g NNO_2^-/L	g NNO_3^-/L	g total N/L
0	0	0.009	0.003	0.279	0.292
24	2.4	0.009	0.003	0.299	0.311
48	4.8	0.011	0.003	0.266	0.280
50.33	5.033	0.009	0.003	0.252	0.265
51.75	5.175	0.014	0.002	0.253	0.269
54.75	5.475	0.016	0.002	0.26	0.278
57.25	5.725	0.016	0.002	0.256	0.274
71	7.1	0.015	0.003	0.283	0.301