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TECHNICAL NOTE: 83.8

EXPERIMENTAL TESTS PROTOCOL FOR RESEARCH OF THE REACTOR OPTIMAL YIELD

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Change of the title to be in accord with the terms of the contract	Cover page and page bottoms	
Change of EPAS TN number to be in accord with the TN number emitted by EPAS	1 & 2	Introduction
Explanation of the term 'standard'	1 & 2	Introduction

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1. INTRODUCTION

At the Paris ESA HQ meeting on 22 April 2005, it has been decided to look for optimisation of the 100 litres pilot reactor by acting on the 'liquid' (and 'solid', eventually) residence time. Before applying the tests on the reactor itself, a protocol must be designed on the basis of the LGCB model (dated on 30 July 2004). It is the aim of the study.
In order to fit the simulation of the 100 litres pilot reactor, the previous simulator, described in TN 71.8.1 for the 25 litres prototype reactor, has been modified so that to keep unchanged the residence time. So as the liquid volume is multiplied by 4 (from 25 to 100 litres), the input liquid flow is also multiplied by the same ratio of 4 : the liquid input flow rate is set to 0.315 l/h (instead of 0.07875 l/h).

For practical reasons, this operating point is called 'standard' in the present study. It is emphasized that it is not connected with any nominal operating point of the pilot plant.

Definition of the standard point :
The input flow rate is equal to 0.315 l/h. The drain flow rate is considered here as a degree of freedom and is determined in function of the objectives. The filtrate flow rate is the difference between the input and the drain flow rates so that the liquid volume remains constant at its nominal value of 100 litres.
Table 1 gives the standard input concentrations. The standard load is the product of these concentrations and of the standard input flow rate (Table 2).

Compound	Input concentration (g/l)
Faeces	3.968
Wheat	7.936
Salad	7.936
Potato	7.936
AA	0.091
PA	0.018
BA	0.021
VA	0.006
CA	0.007
NH_3	0.025

Table 1 : Input concentrations of the standard operating point.
The concentrations of the dissociated compounds are given for the total form.

Compound	Standard load (25 litres prototype)		Standard load (g/h) (100 litres pilot)	
	(g/h)	(g/d)	(g/h)	(g/d)
Faeces	3.1248e-001	7.5	1.25	30
Wheat	6.2496e-001	15	2.5	60
Salad	6.2496e-001	15	2.5	60
Potato	6.2496e-001	15	2.5	60
AA	7.1662e-003	0.172	2.8665e-002	6.8796e-001
PA	1.4175e-003	0.034	5.6700e-003	1.3608e-001
BA	1.6538e-003	0.040	6.6150e-003	1.5876e-001
VA	4.7250e-004	0.0113	1.8900e-003	4.5360e-002
CA	5.5125e-004	0.0132	2.2050e-003	5.2920e-002
NH_3	1.9688e-003	0.0472	7.8750e-003	1.8900e-001

Table 2 : Standard loads of the 100 litres reactor (and of the 25 litres one for the record). The loads of the dissociated compounds are given for the total form.

The corresponding standard residence time is 317 h (13 days) for both reactor (prototype and pilot).

Methodology

The drain flow rate has to be fixed. It will be done by the choice of the ‘Total Solid’ concentration setpoint.

The first step is to look for the yield in function of the ‘Total Solid’ concentration at a given residence time. As it will be seen in next section, the yield is ascending versus the ‘Total Solid’ concentration (the higher concentration, the better yield).

So the study of the yield versus the residence time will be done at maximum ‘Total Solid’ concentration, which will determine the drain flow rate. In section 2 of TN 71.8.1, dedicated to specifications, the maximum constraint of ‘Total Solid’ is fixed to 45 DMg/l.

2. YIELD VERSUS TOTAL SOLID CONCENTRATION

In this section the yield has been computed in function of the 'Total Solid' concentration (in the reactor) at steady state for the standard inputs described in section 1.

Definitions :

1. Yield η :

$$\eta = \frac{v}{u} \quad \text{(dimension less)} \qquad (1)$$

with v : (g/h) sum of production rates of VFA and CO_2
u : (g/h) load (all the input compounds, except water, going into the reactor).

2. 'Total Solid' concentration :

It is the sum of concentrations of all the solid compounds (Faeces, Wheat, Potato, Salad, OMProt, OMLip, OMCarb, OMFibre, BioSugar, BioSugar2, BioAA, BioLCFA, BioDead, SolidInert).

Figure 1 shows the evolution of the yield in function of the 'Total Solid' concentration in the reactor.

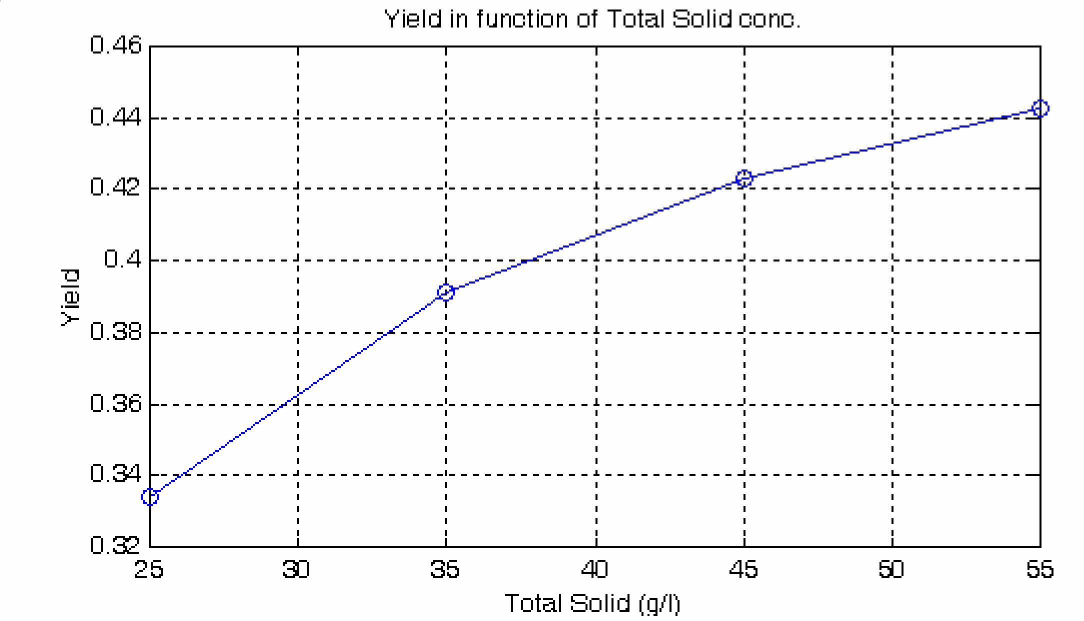


Figure 1 : Yield (dimension less) in function of 'Total Solid' concentration in the reactor.

The result of figure 1 is considered general : the yield is ascending versus the 'Total Solid' concentration in the reactor. So for the next step of the study the 'Total Solid' concentration setpoint is set to its maximum constraint : 45 DM g/l.

3. YIELD VERSUS 'LIQUID' RESIDENCE TIME

In this section :

- The liquid residence time is variable by moving the input liquid flow rate. The input concentrations vary in the inverse way so that the load remains at its standard value defined in table 2 for the 100 litres reactor.
- the drain flow rate is computed so that the 'Total Solid' concentration is equal to its maximum constraint : 45 DM g/l.

The evolution of the yield is given in figure 2. It is plotted under different forms for a better description. The upper graph shows that the yield of the reactor as defined in relation (1) is decreasing versus the liquid residence time. In the two graphs below, the yield is decomposed into its 2 main components :

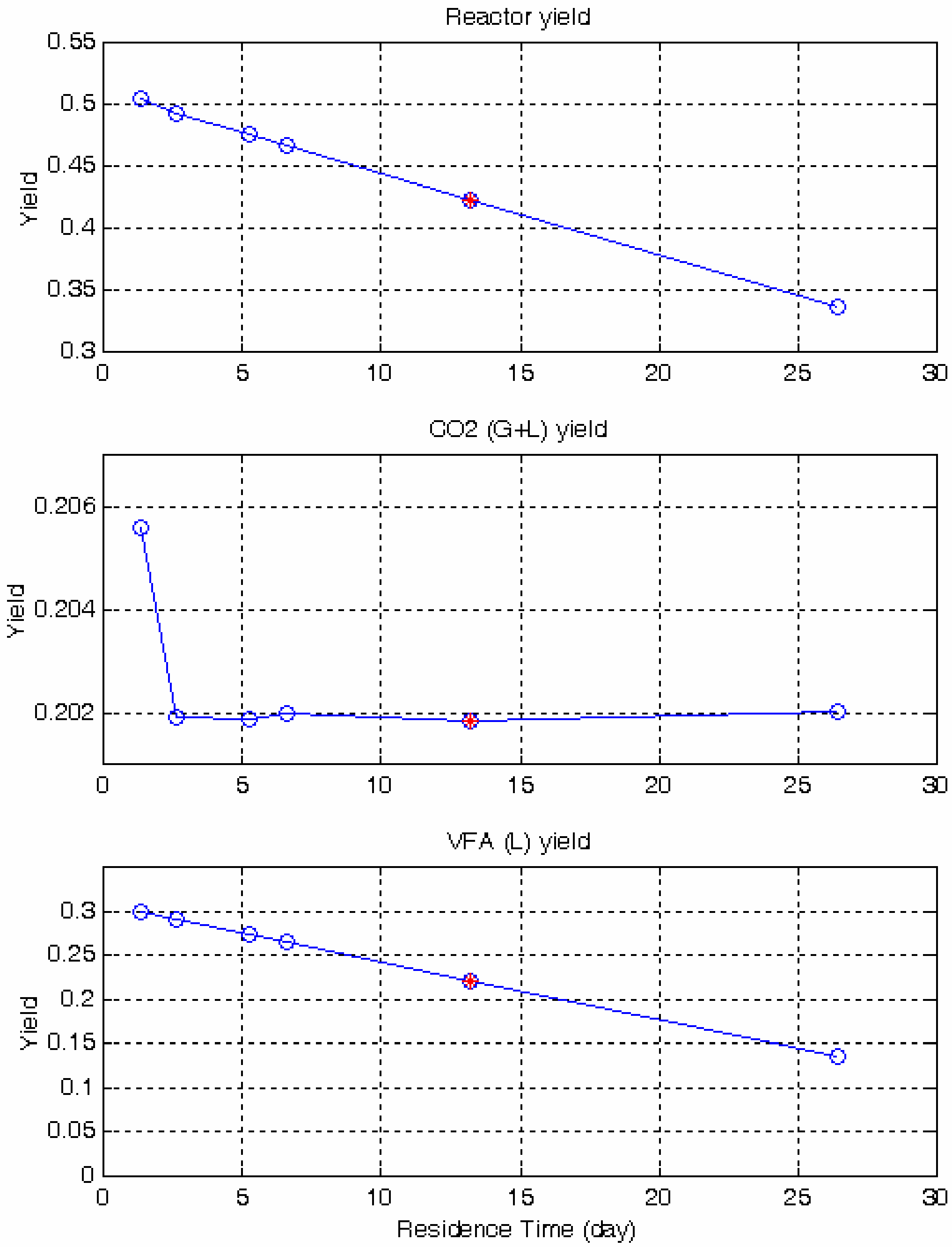
- the yield of CO_2 total in gas and liquid flow rates;
- the yield of VFA that are present in the liquid only.

The yield of CO_2 total in gas and liquid is decreasing for short liquid residence time and then reach an asymptote independent of the liquid residence time while the yield of VFA is continuously decreasing. On the bottom graph of figure 2 it can be extrapolated that the yield of VFA reaches 0 for a residence time of 46 days. In that point the input flow rate is equal to the drain flow rate and all the VFA are lost in the drain. An attempt of explanation is given in relations (A1.22) and (A1.23) for CO_2 and in (A1.15) for VFA.

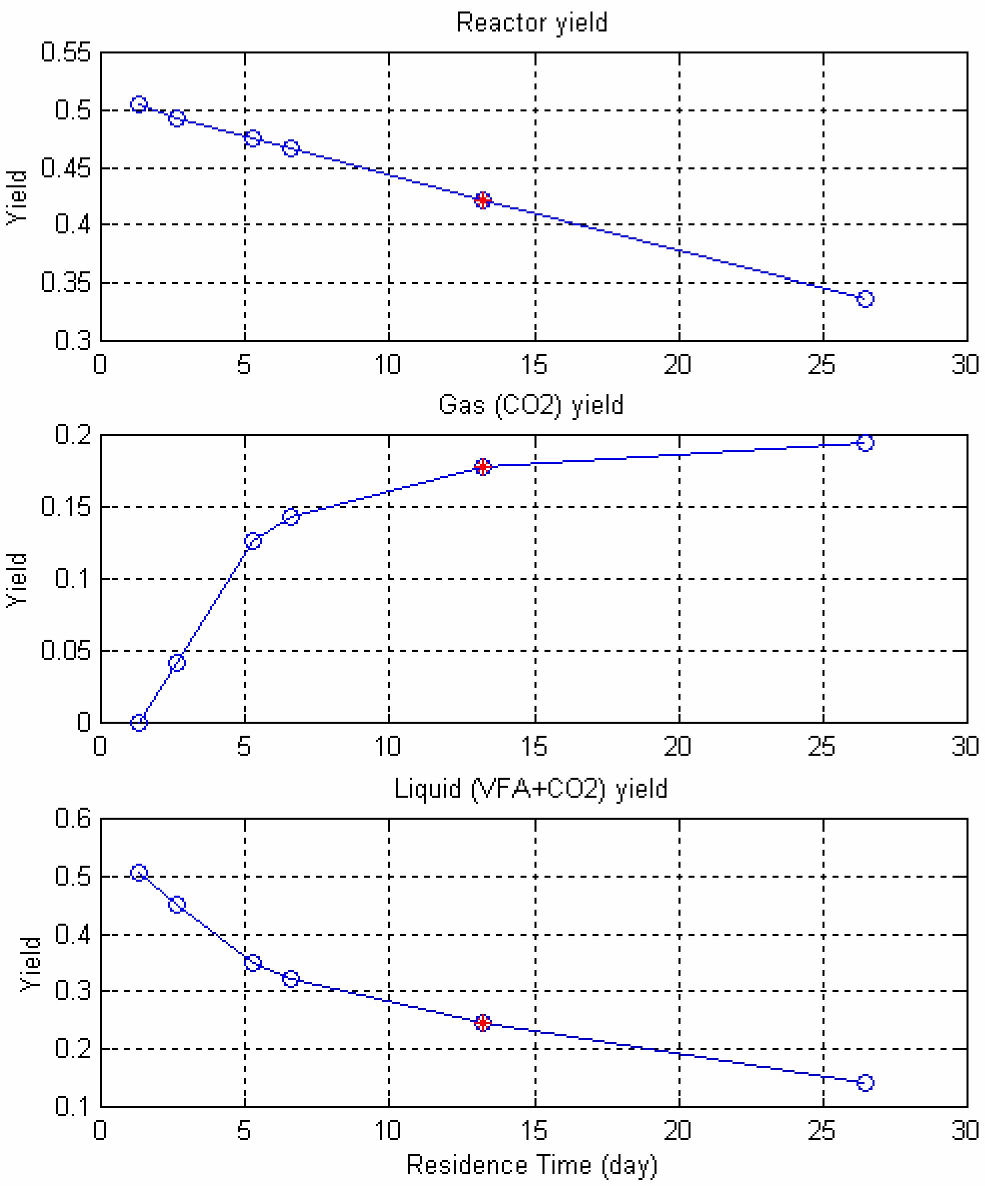
The two lower graphs of the next figure 3, shows another decomposition of the reactor yield :

- the yield in the gas flow that contains CO_2 only;
- the yield in the liquid flow that contains VFA and CO_2.

These two components have inverse behaviour : when the yield in the gas flow is increasing, the other one is decreasing. An attempt of explanation is given in relations (A1.25) and (A1.26) for CO_2.

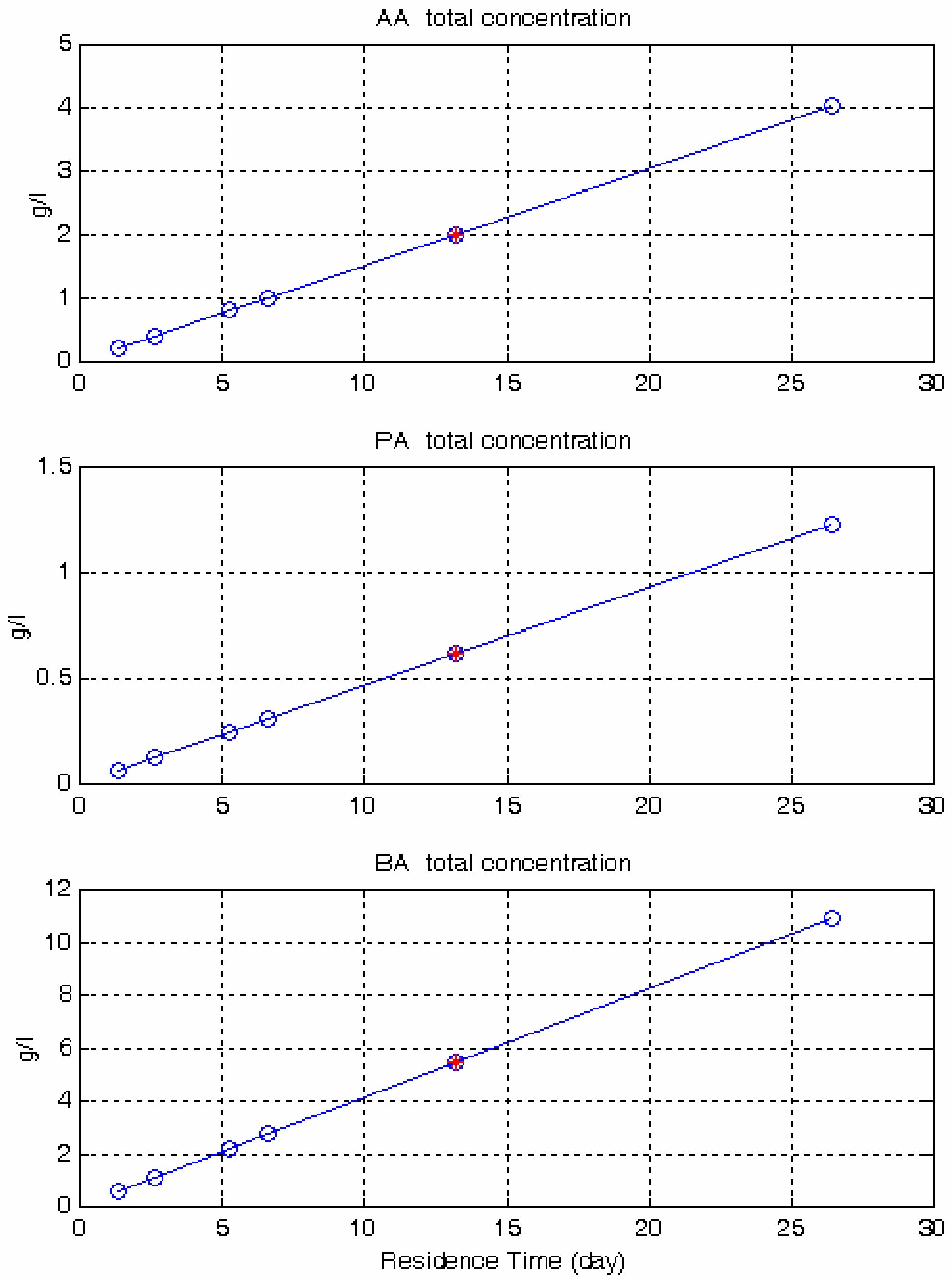


<u>Figure 2</u> : Yield (dimension less) in function of liquid residence time.
The standard point is marked with a red star (at residence time = 13 days).
The reactor yield (upper graph) is decomposed into its two complementary components :
the CO_2 Gas and Liquid component yield
the VFA (Liquid only) component yield



<u>Figure 3</u> : Yield (dimension less) in function of liquid residence time.
The standard point is marked with a red star (at residence time = 13 days).
The reactor yield (upper graph) is decomposed into two components :
the Gas (CO_2 only) yield
the Liquid (VFA and CO_2) yield

The evolutions of the VFA, CO_2 and NH_3 concentrations versus residence time are plotted in the figures 4 and 5. It can be checked that all the concentrations are increasing with the residence time, except for the CO_2 liquid that reaches a saturation limit (graph 3 of figure 5). That can be justified by the relation (A1.18) and (A1.19) of annex 1.



<u>Figure 4</u> : Concentrations of VFA (AA, PA and BA) in function of liquid residence time. The standard point is marked with a red star (at residence time = 13 days).

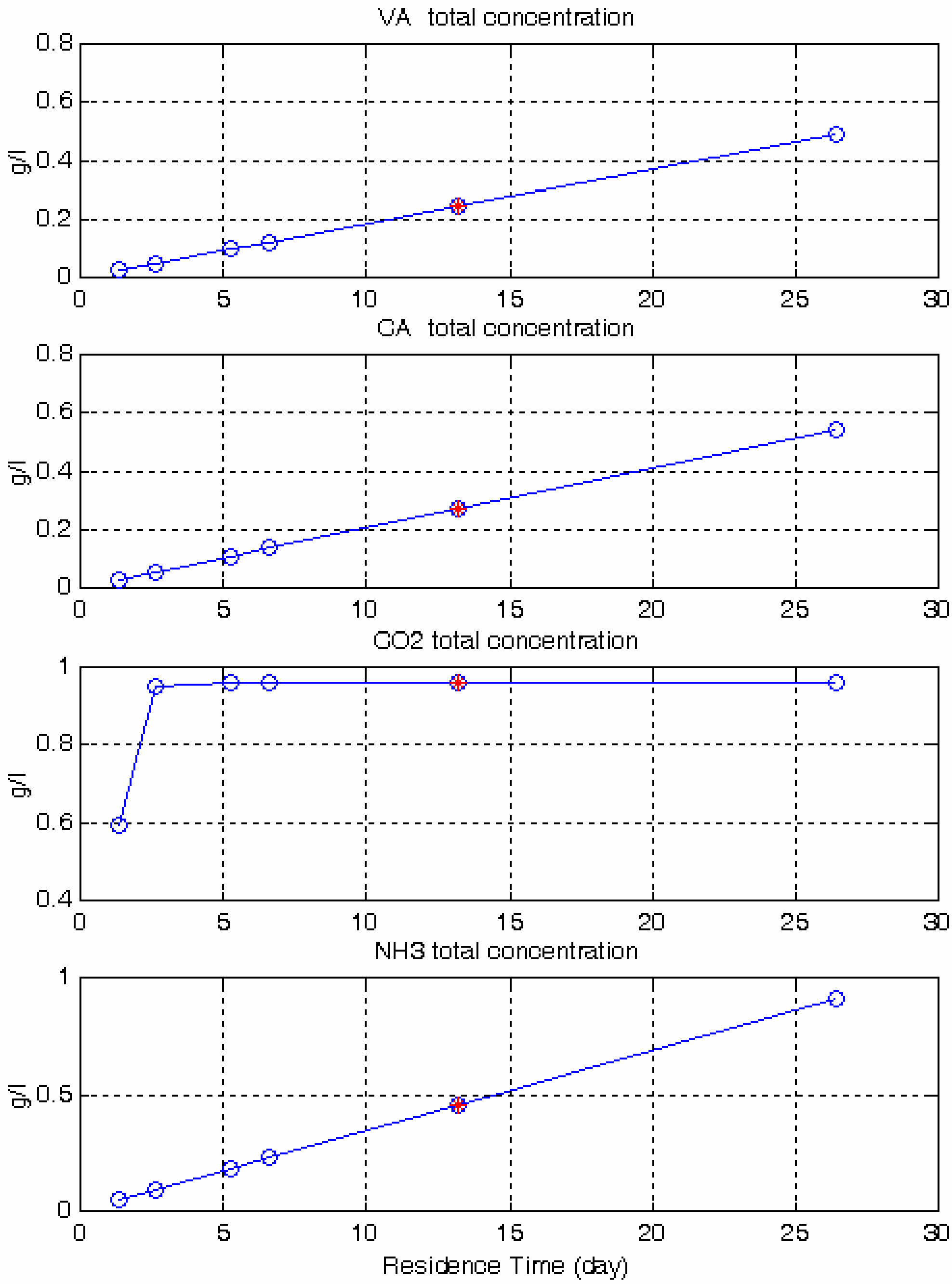


Figure 5 : Concentrations of VFA (VA and CA) CO_2 and NH_3 in function of liquid residence time.
The standard point is marked with a red star (at residence time = 13 days).
In graph 3, the label 'CO_2 total' means CO_2 total form (ionic and molecular) in *liquid*.

As a subsidiary result, it appears that the drain flow rate at steady state is independent of the residence time (figure 6). An attempt of justification is also given in relation (A1.10) of annex 1. So, as the 'Total Solid' concentration is maintained at its maximum constraint (45 DM g/l), the 'Total Solid' production is independent of the residence time and cannot be optimised.

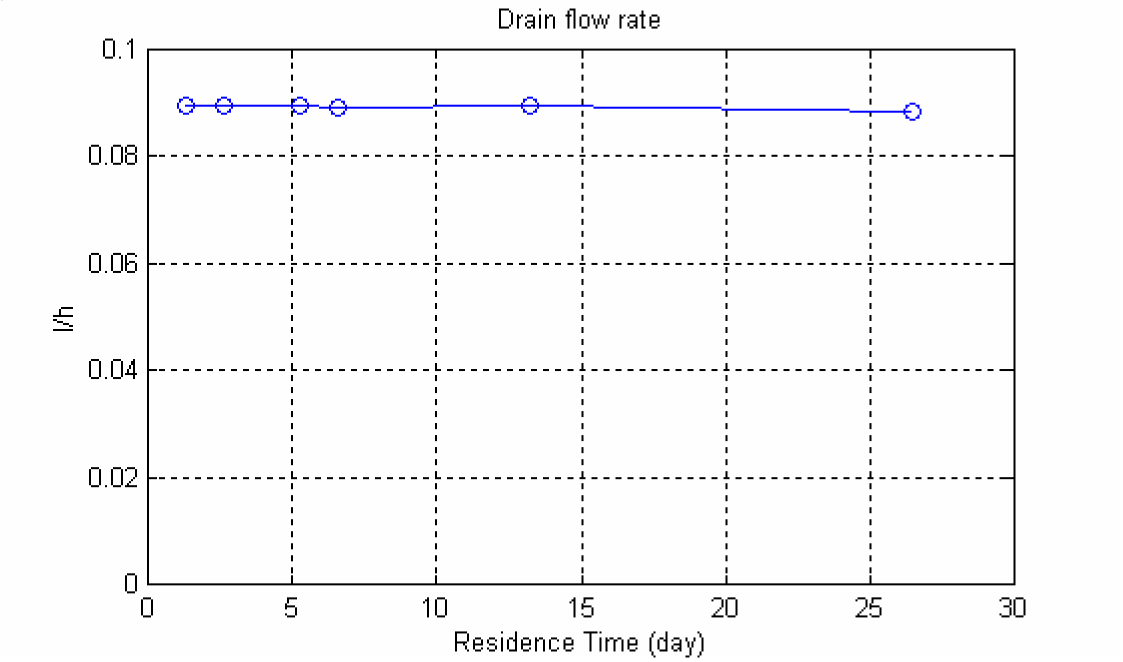
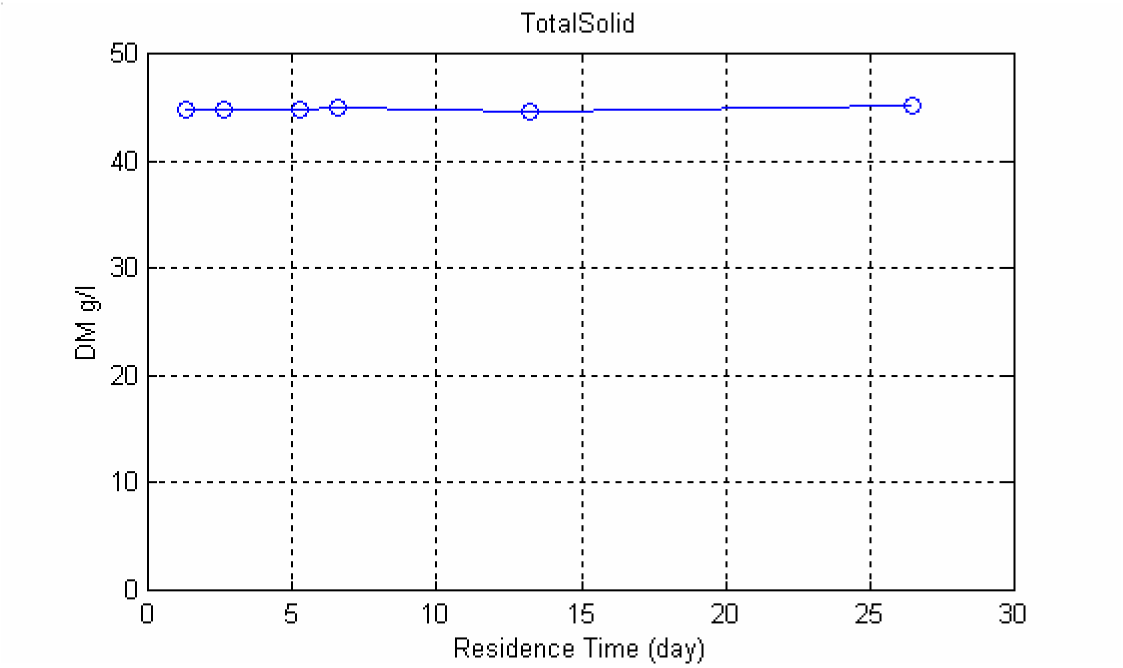


Figure 6 : **Steady state drain flow rate in function of 'liquid' residence time.**

Figure 7 allows to check that the 'Total Solid' constraint (45 DM g/l) is respected.



<u>Figure 7</u> : Steady state ‘Total Solid’ in function of ‘liquid’ residence time.

4. CONCLUSION AND PROPOSED PROTOCOL

The present study shows that :

- the yield of the modelled reactor is decreasing versus ascending residence time;
- the component of the yield related to the total CO_2 in gas and liquid is quickly decreasing and then reaches an asymptote independent of the residence time;
- the component of the yield related to the VFA is continuously decreasing versus ascending residence time. It tends to 0 when the residence time tends to 46 days; in that point, the input liquid flow rate is equal to the drain flow rate and all the VFA are lost in the drain.
- the component of the yield related to the gas production (CO_2 only) is increasing versus ascending residence time and reaches an asymptote for high residence time (about 46 days);
- the component of the yield related to the liquid production (VFA and CO_2) is decreasing versus ascending residence time.

Surprisingly that behaviour can be justified by means of two very simple examples based on the dissolution of a solid into a solvated compound with or without degassing. These examples are studied analytically in annex 1. Further investigations could be done by making more complex these two examples by introducing several solids and several products so that to go towards the complexity of the model. It is expected to express the concentrations of the products, and consequently the yield, at steady state.
When doing the tests proposed hereafter, it should be interesting to check if the pilot yield behaves as foreseen by the model. Then correlations between analytical results and measurements on the reactor could lead to estimate kinetics constants.

Protocol of test to search optimum yield :
The test should cover a large range of residence time to show clearly the evolution of the yield despite the noise measurement.

So it is proposed to realise three tests at the following residence times : 3, 7 and 13 days. During these tests, attention will be focused on the concentrations of CO_2 , VFA and NH_3 in the filtrate and on the production of CO_2 gas. Each test should be long enough (about 4 or 5 residence times) to observe the steady state where the liquid concentrations become constant versus time. The yield and its different components will be measured at steady state. Of course the tests will be done with the same load (the one chosen for the MELISSA project) and the ‘Total Solid’ concentration is supposed maintained at its maximum constraint (45 DM g/l).

According to the present study, the shorter time constant the better yield. The inconvenience is a high dilution of VFA in the filtrate flow, for the shortest residence times (figures 4 and 5). If it is confirmed by the tests, the optimum functioning of the reactor will be a compromise between the yield and the minimum concentrations of VFA acceptable by the Rhodobacter compartment.

Another way to choose the optimum could be the repartition between the gas production and the liquid production. For example, if the CO_2 gas production is more useful for the MELISSA loop than the VFA production, then it could be decided to choose high residence time to obtain high CO_2 gas production and, consequently, poor VFA production.

5. REFERENCE

POUGHON L., "Anareobic Waste Compartment modelling and simulation toolbox ‘AWC_MS’". ESTEC contract, Memorandum of Understanding TOS-MCT/2002/3161/In/CL Dated January 2002, February 2003, TN 74.1.

POUGHON L., "Anareobic Waste Compartment modelling and simulation toolbox ‘AWC_MS’ – AWC toolbox improvement". ESTEC contract, Memorandum of Understanding TOS-MCT/2002/3161/In/CL Dated January 2002, August 2003, TN 74.2.

BRUNET J., LECLERCQ J.J., “Simplified Model”. EPAS Contract dated April 2004, Memorandum of Understanding TOS-MCT/2002/3161/In/CL Dated January 2002, April 2005, TN 1.

BRUNET J., LECLERCQ J.J., “Technical specifications of the Control Command System”. EPAS Contract dated April 2004, Memorandum of Understanding TOS-MCT/2002/3161/In/CL Dated January 2002, April 2005, TN 2.

6. ANNEX 1 : JUSTIFICATION OF THE YIELD VERSUS RESIDENCE TIME

The behaviour of the yield versus residence time observed on the modelled liquefying compartment can be justified by means of the two following simple processes that must be considered as academic examples.
The first example can be seen as the dissolution of a solid into water with a non null rate constant and a first order kinetics. It is assumed to be a very simple representation of the production of VFA from wastes.
In the second example, a degassing is associated to the dissolution. Then it is assumed to represent the production of CO_2 gas.

6.1. First process : dissolution without degassing

6.1.1. DYNAMIC BEHAVIOUR

The input substrate A (concentration 'a_i' and flow rate 'q_i') is supposed to be a solid that is degraded into a soluble product inside a CST (Continuous Stirred Tank) reactor fitted with a filtration unit (figure A1.1).

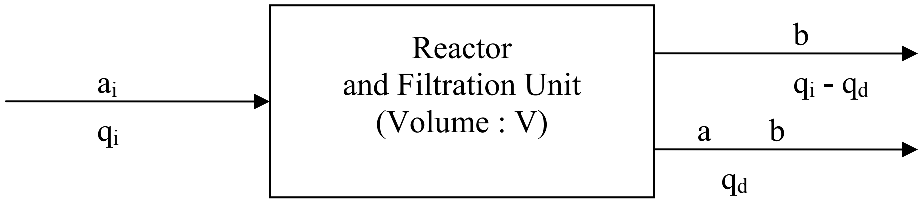


Figure A1.1: Scheme of a simple degradation process
q_i : input flow rate
q_d : drain flow rate
a : concentration of A in the drain flow
b : concentration of product B in the filtrate flow and drain flow
Volume: V=constant

The chemical reaction is supposed to be described by the stoechiometry:

$$\alpha A \rightarrow \beta B \qquad \text{with } \alpha = \beta = 1$$

The process behaviour is described by the following equations:

- Dilution of A and B :

$$V \cdot \frac{da}{dt} = q_i \cdot a_i - q_d \cdot a + V \cdot r_A \qquad (A1.1)$$

$$V \cdot \frac{db}{dt} = -q_i \cdot b + V \cdot r_B \qquad (A1.2)$$

- Kinetics (the reaction is supposed of first order):

$$\frac{d[A]}{dt} = -\mu \cdot [A] \quad \Leftrightarrow \quad r_A = -\mu \cdot a \qquad (A1.3)$$

- Stoechiometry:

$$-\frac{1}{\alpha} \cdot \frac{d[A]}{dt} = \frac{1}{\beta} \cdot \frac{d[B]}{dt} \quad \Leftrightarrow \quad -\frac{1}{\alpha} \cdot r_A = \frac{1}{\beta} \cdot r_B \qquad (A1.4)$$

with r_A : degradation rate of A.
μ : rate constant

Expression of the solid compound concentration dynamic behaviour :
Combining (A1.1) and (A1.3) leads to:

$$\boxed{\begin{array}{lll} \dot{a} = -\frac{1}{\tau_1} a + \frac{G_1}{\tau_1} \cdot a_i \quad \Leftrightarrow \quad \frac{a}{a_i} = \frac{G_1}{1 + \tau_1 \cdot p} & & \text{(in Laplace notation)} \\ \text{with} \quad G_1 = \frac{q_i}{q_d + V \cdot \mu} & & \\ \qquad\quad \tau_1 = \frac{V}{q_d + V \cdot \mu} & & \end{array}} \qquad (A1.5)$$

Expression of the solvated compound concentration dynamic behaviour :
Combining (A1.2) and (A1.3) leads to:

$$\dot{b} = -\frac{q_i}{V} \cdot b + \frac{\beta}{\alpha} \cdot \mu \cdot a \qquad (A1.6)$$

Combining (A1.2) , (A1.3) and (A1.5) leads to:

$$\frac{b}{a_i} = \frac{G_2}{(1+\tau_1 \cdot p)\cdot(1+\tau_2 \cdot p)} \quad \text{(in Laplace notation)}$$

$$\text{with} \quad G_2 = \frac{\beta}{\alpha}\cdot\frac{V\cdot\mu}{q_d + V\cdot\mu} \qquad \text{(A1.7)}$$

$$\tau_2 = \frac{V}{q_i}$$

6.1.2. STEADY STATE BEHAVIOUR

Given the load L :

$$L = q_i \cdot a_i \qquad \text{(A1.8)}$$

It is recalled that the load is constant when q_i varies. The product of q_i and a_i is unchanged.

At steady state (the derivatives are null), the expression of the solid concentration in (A1.5) becomes :

$$a = G_1 \cdot a_i \qquad \text{(A1.9)}$$

The drain flow rate 'q_d' is such that 'a' is equal to its constraint a_M. So (A1.1) becomes :

$$q_d = \frac{L - V\cdot\mu\cdot a_M}{a_M} \quad \Leftrightarrow \quad q_d + V\cdot\mu = \frac{L}{a_M} \qquad \text{(A1.10)}$$

Given the residence time :

$$\tau = \frac{V}{q_i} \qquad \text{(A1.11)}$$

As q_d must be between 0 and q_i, the following inequalities are deduced :

$$A \le 1 \quad \text{with} \quad A = \frac{V\cdot\mu\cdot a_M}{L}$$

$$\tau \le \tau_M \quad \text{with} \quad \tau_M = \frac{V}{L}\cdot\frac{a_M}{1-A} \qquad \text{(A1.12)}$$

The expression of the liquid compound 'b' at steady state is deduced from (A1.7), taking into account that $\alpha = \beta = 1$,

$$b = \frac{V\cdot\mu}{q_d + V\cdot\mu}\cdot a_i = \mu\cdot a_M\cdot\tau \qquad \text{(A1.13)}$$

So at this point of the example, two observations can be done :

- (A1.10) ⇒ qd is independent of τ. It is quite similar to the evolution of the drain flow rate versus the residence time in the figure 6 of section 3.
- (A1.13) ⇒ b is a linear ascending function of τ. It is quite similar to the evolution of the VFA concentrations versus the residence time in the figures 4 and 5 of section 3.

Computation of the yield

By definition the yield η is :

$$\eta = \frac{b \cdot (q_i - q_d)}{L} \tag{A1.14}$$

Its expression versus τ is :

$$\eta = A \cdot \left(1 - \frac{L \cdot (1 - A)}{V \cdot a_M} \cdot \tau\right) \quad \text{(A given in (A1.11))} \tag{A1.15}$$

So the yield is decreasing versus τ with the negative slope :

$$\frac{d\eta}{d\tau} = -A \cdot \frac{L \cdot (1 - A)}{V \cdot a_M} \quad \Leftrightarrow \quad \frac{d\eta}{d\tau} = -\mu \cdot (1 - A) \tag{A1.16}$$

The yield tends to its maximum $A = \frac{V \cdot \mu \cdot a_M}{L}$:

- when τ tends to 0;
- or/and when A tends to 1 (when A=1, it is independent of τ) : i.e. for increasing values of V, μ and a_M, and decreasing values of L.

When $A \neq 1$, the yield tends to 0 when τ tends to τ_M (defined in (A1.12)).

The behaviour of the yield in (A1.15) is very similar to the reactor yield observed in figure 2 of section 3.

From (A1.13), the concentration ‘b’ of the liquid compound ‘B’ increases with ascending residence time. So, as q_d is constant (from (A1.10)), the loss of B in the drain increases. It is another way to explain why the yield regarding B is decreasing.

In the following example, the compound B is assumed to have a gaseous form. Then its yield will be completely different.

6.2. *Second process : dissolution with degassing*

6.2.1. DYNAMIC BEHAVIOUR

In addition to the previous example, the compound B here is supposed to be in equilibrium with its gaseous phase. Then the expression of ‘b’ from (A1.2) becomes :

$$V \cdot \frac{db}{dt} = -q_i \cdot b + V \cdot \phi + V \cdot r_B$$

$$\phi = K \cdot (c - b) \quad \text{with } K = K_{La} \qquad \text{(A1.17)}$$

$$c = \frac{n_0}{k_p} \cdot \frac{n}{n + n_{N2}} \cdot M$$

with n0 = 55.55 mol/l,

kp : partition coefficient of compound B,
M : molar mass of compound B.
ϕ : positive in the direction from gas to liquid
n : number of mole of B in the gas phase.
n_{N2} : number of mole of nitrogen (inert gas) in the gas phase.

Remark : the expression of 'c' above tend to its asymptote when the n increases while n_{N2} is constant with the time :

$$c_A = \frac{n_0}{k_p} \cdot M \qquad \text{(A1.17 bis)}$$

6.2.2. STEADY STATE BEHAVIOUR

At steady state (the derivative is null) and the expression of 'b' from (A1.17) becomes :

$$b = \frac{\mu \cdot a_M + K \cdot c}{K + \frac{1}{\tau}} \qquad \text{(A1.18)}$$

So when τ tends to 0, b tends to 0,
and when τ tends to τ_M (equivalent to ∞), b tends to its asymptotic value :

$$b_A = \frac{\mu \cdot a_M + K \cdot c}{K} \qquad \text{(A1.19)}$$

This behaviour is very similar to the one observed for liquid CO_2 (total form) concentration in graph 3 of figure 5, section3.

Computation of the yield

In the case of a compound B present in gas and liquid phases, the definition of the yield η is :

$$\eta = \frac{-\phi \cdot V + b \cdot (q_i - q_d)}{L} \qquad \text{(A1.20)}$$

Its expression versus τ is :

$$\eta = \frac{1}{L} \cdot \left(V \cdot \mu \cdot a_M - q_d \cdot \frac{\mu \cdot a_M + K \cdot c}{K + \frac{1}{\tau}} \right) \qquad \text{(A1.21)}$$

So when τ tends to 0, the yield tends to its maximum :

$$\eta_M = \frac{V \cdot \mu \cdot a_M}{L} \leq 1 \qquad \text{(A1.22)}$$

and when τ tends to τ_M (equivalent to ∞), the yield tends to its asymptote :

$$\eta_A = \frac{1}{L} \cdot \left(V \cdot \mu \cdot a_M - q_d \cdot \frac{\mu \cdot a_M + K \cdot c}{K} \right) \qquad \text{(A1.23)}$$

It is similar to the behaviour of CO_2 (Gas + Liquid) yield in figure 2 of section 3.

Gas component of the yield :

Considering the gas component of the yield :

$$\eta_G = \frac{-\phi \cdot V}{L} \qquad \text{(A1.24)}$$

its expression is :

$$\eta_G = \frac{K \cdot V}{L} \cdot \left(\frac{\mu \cdot a_M + K \cdot c}{K + \frac{1}{\tau}} - c \right) \qquad \text{(A1.25)}$$

When τ tends to 0, ‘c’ tends to 0 because all the gas remains in the liquid phase; so η_G tends to 0;

and when τ tends to τ_M (equivalent to ∞), the component η_G of the yield tends to its asymptote :

$$\eta_G = \frac{K \cdot V}{L} \cdot \left(\frac{\mu \cdot a_M + K \cdot c_A}{K} - c_A \right) \qquad \text{(A1.26)}$$

It is similar to the behaviour of Gas (CO_2) yield in figure 3 of section 3.

Liquid component of the yield :

In this example where the compound B can be in a gaseous form, its concentration ‘b’ in the liquid phase reaches a maximum asymptotical value (from (A1.19)). So its loss in the drain is limited, contrarily to what happens when B has no gaseous form (case of the previous example).

NOTATIONS

CODs	Soluble Chemical Oxygen Demand
CODt	Total Chemical Oxygen Demand
DM	Dry Matter
N org	Organic Nitrogen
N tot	Total Nitrogen
OM	Organic Matter
VFA	Volatile Fatty Acids
AA	Acetic Acid (CH_3COOH)
BA	Butyric Acid (C_3H_7COOH)
CA	Caproic Acid ($C_5H_{11}COOH$)
PA	Propionic Acid (C_2H_5COOH)
VA	Valeric Acid (C_4H_9COOH)
NH_3	Ammonia (gaseous or solvated)
CST	Continous Stirred Tank
ODE	Ordinary Differential Equation
MV	Manipulated Variable
Sp	Setpoint
CCS	Control Command System

7. ANNEX 2 : DOCUMENT EVOLUTION

	ESA Comment	Sherpa answers
Cover page	Could you please change the title of the TN so that it reflect the WP name? In this case, it should be "Support to testing of the system model of CI"	According to the corresponding WP title of Sherpa proposal, the title should be "Experimental tests protocol for research of the reactor optimal yield"
Introduction, page 1 and 2	Could you please change the number of the SHERPA TNs you refer to? (i.e. "SHERPA's TN 1, page 1, TN2 page 2) These SHERPA TNs have been combined by EPAS in TN 71.8.1	Done accordingly
	Could you please clarify the way the input flow rates are calculated? Nominally, the hydraulic residence time in the waste compartment is 10 days, which implies that for the 100L reactor, 10 L influent are fed every day. Flow rates may be higher. Could you please check if there is an impact on the results depicted in the TN?	The term 'standard residence time' refers to a given operating point in the present TN. This point is defined in introduction. It is not connected with the nominal operating point of the pilot plant.