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FLOW INVESTIGATION IN AN INNOVATING DYNAMIC FILTRATION MODULE USING TRACING METHODS

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ABSTRACT

Residence Time Distribution (RTD) experiments were carried out under laminar and turbulent
regimes in a complex dynamic filtration module, named Rotating and Vibrating Filtration (RVF).
This filtration module, dedicated to bioprocess intensification and downstream processing,
consists of two filtration cells in series in which a three-blade impeller rotates between two flat
membranes.

48 Our objectives are to improve filtration and overall industrial bioprocess performances by (i) 49 deeply understanding the flow behaviour within RVF modules and (ii) characterizing and 50 modelling the RTD through a systemic analysis and (iii) identifying critical operating conditions 51 with microbial cells.

Analytical study of distribution functions was conducted and statistical moments were calculated and discussed. This study provides useful recommendations, guidelines by identifying efficient volume (functioning area), dead zone volume (dysfunctioning area). The influence of operating parameters (mixing rate N and flow rate Q_f) on the mean residence time, t_s were highlighted. The systemic analysis led to compare three models with analytical solutions. Finally, a simple model allowing the description of the evolution of the RTD of the studied filtration module was proposed.

59 Keywords:

Dynamic filtration; thermal balance, Residence time distribution; modelling; systemicapproach.

62

63 Nomenclature:

Latin Letters		
С	Concentration (salt)	[g/L]
C _p	Specific heat capacity	[J/ (kg·°C)]
	Impeller diameter	[m]
Ea	Activation energy	[kJ/mol]
E(t)	Residence time distribution function	[1/s]
F(t)	Cumulative distribution function	[/]
J	Number of reactors	[/]
N	Mixing rate	[Hz]
N _p	Power consumption number	[/]
Pe _L	Peclet number	[/]
Q_f	Feeding flowrate	[L/h]
R	Universal gas constant, 8.314 J/(mol °C)	[J/(mol °C)]
Re _{mixing}	Reynolds number of a mixing system	[/]
Re _Q	Reynolds number of a system equivalent to a tube	[/]
t _s	Mean residence time	[s]
Т	Temperature	[°C]

ТМР	Transmembrane pressure	[bar]
<i>V</i> ₁ , <i>V</i> ₂	Used volume and dead-zone volume, $V_1+V_2=V_{RVF}$	[L]
V _{RVF}	Total volume of RVF module, 1.47 L	[L]
x(t), y(t)	Experimental inlet and outlet reduced and normalized signals	[1/s]
X(t), Y(t)	Cumulative function of $x(t)$ and $y(t)$	[/]
X(s), Y(s), G(s)	Laplace transform function of $x(t)$, $y(t)$ and $E(t)$	[/]
Greek symbols		
μ	Viscosity	[Pa·s]
ρ	Density	[kg/m ³]
β^2	Reduced variance (centred moment of 2 nd order)	[/]
τ	Holding time	[s]
Γ	Centred moment of the given order	
α	Slope (regression coefficient)	
σ	Electric conductivity	[S/m]
Abbreviations		
CF	Cross-flow	
CFD	Computational Fluid Dynamic	
CSTR	Continuous stirred-tank reactor	

DF	Dynamic Filtration	
DE	Dead-end (filtration)	
OVL	Overlapping coefficient,	
PF	Plug flow reactor	
RVF	Rotating and Vibrating Filtration module	

66 1 Introduction

The principle of Dynamic Filtration (DF) is to generate complex hydrodynamic 67 68 perturbations (magnitude of velocity and shear stress, time dependent) by mechanical movement 69 (rotation, vibration, oscillation) of membrane or external mechanical forces close to the 70 membrane, to better control membrane fouling. The external force applied to the system can be 71 longitudinal, transverse, torsional, or with a mix of these motions. In the recent decades, many 72 efforts have been done to study hydrodynamics and to develop novel DF modules at lab scale or 73 pilot scale. In each device, the controls of local and global performances (permeability, fouling, 74 limitations) are closely related to our knowledge of local and global hydrodynamic. The existing 75 DF modules can be classified in terms of mechanical movement as cylindrical rotating filter 76 (such as *Biodruck-filter*[1], *Biopurification System*[2], *RDF filter*[3]), rotating flat membrane 77 filter (such as CRD filter[4], MSD filter[5], RDM module[6-8], DYNO filter[9], RVF module[10-78 12]), and vibrating filter (such as VSEP filter[13], VMF filter[14] and hollow fibber filter[15,16]). 79 Compare with dead-end (DE) and cross-flow (CF) filtration, DF has been proved (1) by reducing 80 environmental impact (low loop volume), (2) by reducing energy consumption, uncoupling 81 between the conventional driving force (from feeding flowrate) and wall shear stress, working 82 under low transmembrane pressure (TMP). Enhancements in DF are mainly attributed to 83 complex hydrodynamics near the membrane surface due to the various motions (rotation, 84 vibration, oscillation) of the external driving forces.

Recent development in the processing and technology of dynamic filtration have motivated numerous of researches in the evaluation of membranes filters quantitatively and qualitatively, in the domain of drinking/waste water treatment, food engineering, pharmacy and biological processing and so on. Basically, the accumulation of material rejected and remained on or near

89 the membrane surface always leads to a decline of permeate flux over time. Many efforts have 90 been done to recover or maintain a high efficiency and quality of permeate by using plenty of 91 low viscosity feeding fluid. However, less attention has been paid to the fluid transport and flow 92 behaviour in the system, less is known about the homogeneity which might be affected by the flow perturbation and mixing, both in laminar and turbulent regime. To provide systemic 93 94 information, Residence Time Distribution (RTD) is a crucial approach to diagnose the flow 95 performance associated with degree of mixing and shearing which play an important role in the final product quality[17]. 96

97 Therefore, in this paper, investigation of RTD was performed in a pilot plant with a 98 dynamic filtration module integrated in an instrumented open loop. As a response variable, 99 thermal balance and RTD are crucial parameters that has been commonly used to investigate the 100 performance of fluid mixing and to diagnose the defect of a system design. Our objective was: 101 (1) to study the impact of processing conditions (flowrate, rotation speed) on RTD in an 102 industrial pilot-plant (RVF); (2) to compare through distribution functions the RVF 103 hydrodynamic behaviours in laminar and turbulent regimes; (3) to evaluate the homogeneity 104 within the device and (4) to validate a reactor model to predict RTD. In the first step, tracer 105 experiments were performed and scrutinized with different process conditions (flowrate, mixing 106 rate) with Newtonian fluids, embracing laminar and turbulent regimes. Experimental data were 107 interpreted thru the sets of moment and centred moments deduced from distribution functions. 108 Their statistical deviation and evolution were discussed as a function of the process parameters. 109 In a second step, a systemic analysis led to the identification of a suitable model thanks to the 110 comparison of three different models.

111 2 Materials and methods

112 2.1 Filtration module

114 The DF module, shown in fig. 1, is called Rotating and Vibrating Filtration (RVF 115 technology – patent no. FR-97-14825) [18]. It is designed for biological or food liquids. RVF 116 module has been studied in the previous studies [10-12,19], it consists of two filtration cells in series (4 membranes, filtration area = 0.048 m^2 , cell volume = 0.2 L and RVF volume 1.48 L), 117 118 and in each cell, a three-blade impeller (flat blade, $d_m = 138$ mm, thickness = 8 mm) is driven by 119 a central shaft continuously rotating (up to 50 Hz) in a 14 mm gap between two porous flat 120 support (membrane support) which drains the permeate. It gives a 3 mm gap between the 121 impeller and the membrane surface. This simple mechanical device runs continuously and 122 generates a high shear rate as well as a hydrodynamic perturbation in the small gap, TMP (up to 123 300 kPa) and rotation frequency can be adjusted to optimize the operating conditions.

124

113

(figure 1)

Friction and power consumption curves of RVF module were established previously [10], aimed to identify the flow regime of given operating conditions. The critical Reynolds numbers can be obtained to define flow regime as follows: laminar regime: $Re_{mixing} < 1 \times 10^3$; transition regime: $1 \times 10^3 < Re_{mixing} < 3 \times 10^4$; and turbulent regime: $Re_{mixing} > 3 \times 10^4$.

129 2.2 Experimental setup for RTD

Figure 2 shows the experimental set-up which was implemented with: (1) an open loop configuration including a pump (TUTHILL CO., series A54739, Drive: GROSCHOPP CO., series PM8014, 3800RPM, DC motor control unit: DART 250G series, $Q_f = 0$ to 400 L/h), a flowmeter (Flowmeter Altometer IFM 1080/6 DN 15mm, precision 5%), a manometer (0-6bar) and a counter-pressure valves, (2) an injection unit for tracer include a septum for syringe injection followed by a static mixer to achieve homogenous concentration and (3) temperature (Pt1000, -20°C/+150°C, precision: ± 0.15 °C) and conductivity measurements (Conducell 4USF-PG325, range: 1-500000 μ S/cm, precision: $\pm 1\%$ /decade and KEMOTRON type 9147 Integral flow, range: 5-2000000 μ S/cm, precision: $\pm 3\%$ /decade) were located before connecting ducts (Diameter = 12 mm, Length= 125 mm) at inlet and outlet of RVF module. The total experimental volume between the conductivity meters is equal to VRVF=1.61 L.

141

(figure 2)

142 In laminar regime, the fluid was stored at room temperature in a 50 L feeding tank and 143 collected into a recycling tank. In turbulent regime, the fluid was not recycled (open loop).

144 2.3 Experimental fluids and tracers

145 The tracer must have several properties as: (1) non-reactive (2) easily detectable (3) 146 properties similar to the fluid in the system (4) completely soluble (5) should not adsorb [20]. 147 The concentration of the tracer is adjusted considering temperature and fluid according to a 148 known function [21]. The selected tracer should not modify the physical properties of the fluid 149 and the hydrodynamic condition in the system. In present work, saline tracer solutions ([NaCl] = 150 100 g/L) were injected with a 5 mL syringe within a short time (<5s) (close to an ideal pulse 151 injection). The inlet and outlet response signals are obtained by measuring electric conductivity 152 and by calculating the equivalent tracer concentration.

153 The RTD experiments were restricted to two Newtonian fluids (BREOX solution and water) 154 for laminar and turbulent regime respectively. A water soluble and viscous transparent 155 Newtonian fluid (BREOX® Polyalkylene Glycol 75 W 55000, BASF) was used to achieve

156 laminar regime, and water was used for the turbulent regime. For BREOX solution, thermophysical properties including viscosity μ [Pa·s], density ρ [kg/m³], heat capacity C_p [J/(kg·°C)] 157 158 were respectively measured with a rheometer (HAAKE Mars III, SN: 4201100100779, Thermo Scientific, Germany, torque range: 10⁻⁸ to 0.2 N·m, mixing rate range: 10⁻⁷ to 4500 RPM), a 159 density meter (DE40, SN: MPK38384, Mettler Toledo, France, range: 10^{-4} to 3 g/cm³ ± 10^{-4} 160 g/cm^3 , 4 to 90°C \pm 0.05°C), and a differential scanning calorimeter (Micro DSC-III, SN: 161 60/50287.06.102, Setaram, France, temperature range: -20 to +120°C, thermal kinetics: 10⁻³ to 162 163 +1.2°C and fluxmeter range: 0.2 μ W to 20 mW, resolution : 40nW). These three properties μ , ρ and C_p are described as functions of temperature T [°C] and mass concentration C [%] (15 to 164 165 45°C) as follows:

$$\begin{cases} Cp = aT + b \\ a = -14.8C^2 + 16.3C - 1.46 (R^2 = 0.974) \\ b = -2.17 \times 10^3 C + 4.30 \times 10^3 (R^2 = 0.972) \end{cases}$$
(1)

$$\begin{cases}
\rho = dT + c \\
c = 0.185C + 1.00 (R^2 = 0.999) \\
d = -9.74 \times 10^{-4}C - 2.58 \times 10^{-4} (R^2 = 0.999)
\end{cases}$$
(2)

$$\begin{cases} ln\mu = -\frac{Ea}{R} \left(\frac{1}{T + 273} - \frac{1}{T_r + 273} \right) + ln\mu_r \\ Ea = -1.80 \times 10^4 C - 1.90 \times 10^4 (R^2 = 0.964) \\ ln(\mu_{20}) = -7.88C^2 + 19.7C - 6.78 (R^2 = 0.999) \end{cases}$$
(3)

where coefficients a, b, c, d are as the functions of C in Eq. (1) and (2); Temperature dependence of μ is fitted with the Arrhenius-type equation, with E_a is the activation energy in kJ/mol, R is the universal gas constant, μ_r is the viscosity in Pa·s at reference temperature, $T_r = 20^{\circ}C$. Then, properties of BREOX solutions are estimated by Eq. (1, 2 and 3). Consequently, a 35% (mass concentration) diluted BREOX solution was chosen as a test fluid 171 (with $\mu = 0.35$ Pa·s, Cp = 3650 J/(kg·°C) and $\rho = 1040$ kg/m³ at T = 20°C). Temperature of the 172 feeding tank was controlled at about 20°C by adjusting the thermostat (room temperature was 173 controlled at 20°C).

Water was used in RTD to achieve turbulent regime. Its thermal – physical properties and their thermal dependency were taken from literature [21]. At 20°C, the values are: density $\rho = 998.2 \text{ kg/m}^3$, specific heat capacity $C_p = 4181.8 \text{ J/(kg} \cdot ^{\circ}\text{C})$ at constant pressure, viscosity $\mu = 1.0 \text{ mPa} \cdot \text{s.}$ (under 100 kPa).

178 2.4 Methodology

179

180 2.4.1 Theory of Residence Time Distribution (RTD)

181 The residence time of an element in a fluid is defined as the time elapsed from its entry into 182 the system until it reaches the exit. The distribution of these times is called the RTD function of 183 the fluid E(t), or E-curve, and represents the fraction of fluid leaving the system at each time 184 [22]. Practically, experimental injection cannot be a perfect and direct signal. In the experiment 185 point of view, normalized signals x(t) and y(t) were defined for inlet and outlet, presents as 186 Eq.(4). The product of the convolution can be replaced in the Laplace domain by a simple 187 product, where X(s), Y(s) and G(s) are the Laplace transform of x(t), y(t) and E(t), as Eq.(5) 188 presents:

$$y(t) = \int_0^t E(u)x(t-u)du \tag{4}$$

189 and

$$Y(s) = G(s) \cdot X(s) \tag{5}$$

190 Practically, x(t) and y(t) are calculated from experimental signal $C_{in}(t)$ and $C_{out}(t)$ 191 respectively, expressed as Eq. 5, and its cumulative function can be obtained as Eq. 6.

$$E(t) = \frac{c_{out}(t)}{\int_0^\infty c_{out}(t)dt} and F(t) = \int_0^\infty E(t)dt$$
(6) and (7)

192 RTD function can be expressed by moments and centred moments of orders Γ [23]. The 193 mean residence time, t_s , is characterized by a moment of first order, Γ^1 . The holding time, a 194 theoretical value τ , is calculated by the volume of the tested system and the feeding flowrate, as 195 Eq.(9) shows. For most of the simple system, t_s and τ are equal indicating a perfect 196 macromixing. However, in some complex system, it is not the case. Knowing *k* defined by 197 Eq.(10), flow behaviour can be diagnosed if the system has a shortcut or a dead zone (volume 198 V_2), effective volume V_1 is defined by t_s .

$$\Gamma^{1} = t_{s} = \int_{0}^{\infty} tE(t)dt and \tau = \frac{V}{Q_{f}}$$
(8) and (9)

199 and

$$k = \frac{t_s}{\tau} = \frac{V_1}{V_{RVF}} \left(V_1 = t_s \times Q_f, V_{RVF} = V_1 + V_2 \right)$$
(10)

The centred moments of second order, $\Gamma^{2'}$, presents the variance σ^2 , as Eq. (11) shows, and its corresponding dimensionless term β^2 , as Eq. (12) shows. The second centred moment is a very important parameter to describe the width of a distribution, ideal plug flow reactor has $\beta^2 = 0$, $\beta^2 > 0$ indicates an axial dispersion, the smaller the value the narrow the distribution curve and the lower the axial dispersion as well [24].

$$\Gamma^{2'} = \sigma^2 = \int_0^\infty (t - t_s)^2 E(t) dt = \int_0^\infty t^2 E(t) dt - t_s^2 \text{ and } \beta^2 = \frac{\sigma^2}{t_s^2}$$
(11) and (12=

205 *2.4.2 Experimental strategy and operating procedures*

Thermal balance and the hydrodynamic study of Residence Time Distribution were conducted according to a defined conditions and procedures. Each experiment was studied in function of the various operating conditions concerning different test fluids, feeding flowrates, and mixing rate of RVF module in both laminar and turbulent regimes.

RTD experiments were conducted according to defined conditions and procedures. Each experiment were conducted in triplicate (or more) in function of the various operating conditions concerning different test fluids (water and Breox 35%), feeding flowrate, Q_f , (25 to 300 L/h) and mixing rate N (0 to 50Hz) in laminar and turbulent regimes. During tracing experiments, fluid properties and operating parameters were stable, inlet fluid temperature was between 17 and-22°C (±0.14°C) under laminar regime and between 13 and 15°C (±0.1°C) under turbulent regime.

217 *2.4.3 RTD: data treatment and analysis*

Tracing experiments were carried out and analysed according to the methodology described by Thereska et al [25]. The experimental distribution functions enable to diagnose the functioning of pilot and industrial processes.

In the first step, a set-up with fluid circulation connected with RVF module and related sensors were conducted (to optimize experiments and quality of the measurements).

In the second step, experiments were conducted with designed experiment protocol and rawdata was acquired.

In the third step, the processing of experimental raw data was performed by smoothing, convolution, background noise reduction, data selection, normalization and standardization. Raw

data (electrical conductivity) was acquired by data logger, expressed as σ_{in} and σ_{out} at 227 temperature T_{in} and T_{out} , were recalculated at T_{ref} (25°C), based on equation $\sigma = \sigma_{25^{\circ}C} + a \cdot$ 228 $T_{25^{\circ}C}^{n} \cdot (T-25)$, with a=0.022 and n=0.93 [26]. Base line was then subtracted and conductivity 229 signal $\sigma - \sigma_0(t)$ was converted into salt concentration, C. Mass balance was established by 230 comparing inlet and outlet signals, $q_{in} = \int_0^\infty C_{in}(t) \cdot Q(t) \cdot dt$ and $q_{out} = \int_0^\infty C_{out}(t) \cdot Q(t) \cdot dt$. 231 Concentration signals were normalized by total amount of tracer as follows: $x(t) = \frac{C_{in}(t)}{\int_0^\infty C_{in}(t)dt}$ and 232 $y(t) = \frac{C_{out}(t)}{\int_{0}^{\infty} C_{out}(t)dt}$, by taking the contribution of injection effect into account; Thereafter, 233 cumulative function can be calculated by $X(t) = \int_0^\infty x(t) dt$ and $Y(t) = \int_0^\infty y(t) dt$. In the case of a 234 235 perfect inlet impulsion, x(t) is a Dirac distribution and distribution function E(t) will be equal to 236 y(t). In the fourth step, moment and centred moments were discussed concerning mean residence 237 time, and RTD curve including dispersion, asymmetry, and spreading. These moments enable to 238 diagnose the behaviour of the reactor as a function of flow and mixing regimes.

239 In the fourth step, a systemic approach is then conducted by software (DTS Progepi, RTD 240 Software Analysis). This software was used to correctly modelling the experimental response to 241 an input of any complex set-up consisted of connected elementary reactors [27]. This step is 242 based on the identification of parameters (hypothesis of a model based on the interconnection of 243 elementary reactors more or less complex) by adjusting the simulated response according to the 244 experimental data. The software realizes optimization by Rosenbrock's method, allowing 245 simultaneously optimize up to 6 parameters in a reduced way, which corresponds to the difference, $\sum_{0}^{NP} [y(t)_{cal} - y(t)]^2$, between the distribution function $y(t)_{cal}$ calculated by the 246 247 model and the experimental curve y(t).

249 **3 Results and discussions**

An analytical study concerning experimental observations is firstly reported for thermal balance and tracing experiments. Then moments are calculated and discussed as a function of mixing regime. Finnally reactor modelling through systemic approach is proposed.

253 *3.1 Thermal balance*

In order to realize thermal balance, power consumption of RVF module was estimated from power consumption curve. The flow is supposed to be incompressible and stationary, and we neglect the gravity neglected. A non-slip condition is imposed on the walls and the inlet speed profile is the same as the outlet profile, then it gives:

$$\Delta P \cdot Q + \iint_{A} \sum_{j=1}^{3} \sum_{i=1}^{3} \tau_{if} n_{i} U_{j} ds + \iint_{A} \sum_{i=1}^{3} (-P) n_{i} U_{i} ds = \iiint_{D} \rho \varepsilon_{V} dv$$
(13)

With 1^{st} term, necessary pump power to drive the fluid in the circulation loop; 2^{nd} term, external viscous forces of by the impeller; 3^{rd} term, external forces of the pressure by the impeller and 4^{th} term, total power dissipation in the field.

261 Moreover, we assume the uniform temperature at the input and output sections and the heat 262 capacity independent of T, heat dissipation can be simplified by effective power:

$$P_{eff} = \iiint_{D} \rho \epsilon_{V} dv = \rho C_{p} (T_{inlet} - T_{outlet}) Q_{f}$$
(14)

Thermal balance (viscous dissipation) is highly correlated to mixing power. As a result, temperature increases significantly with the increase of mixing rate and the reduction of flowrate. For example, as Fig. 3-A shows, in laminar flow (BREOX solution) with $Q_f = 25$ L/h and N=25 Hz, temperature increased 11°C; in turbulent flow (water), with $Q_f = 50$ L/h and N=50 Hz, temperature increased 9°C.

In agreement with theory (Eq.13 and 14), Fig. 3-B shows the variation of heat dissipation and efficient power consumption [W] under different mixing rate in both laminar and turbulent regimes. Due to its higher viscous force (2nd term of Eq. 13) in laminar regime, temperature (as well as heat dissipated) increases faster than in turbulent regime (for example Q_f =50 and 100 L/h). The effective power consumption is estimated from power consumption curve which was established in a previous research [10], Np = $\left(B^{\frac{1}{n}} + \left(\frac{Kp}{Re_{mixing}}\right)^{\frac{1}{n}}\right)^n \cong \frac{Kp}{Re_{mixing}}$, (equation (14)) where n=2, B=0.10, Kp = 520, by knowing mixing Reynolds.

275 In a bioprocess context, maintaining ideal culture temperatures is vital for optimal cell 276 growth. Which temperature is selected will depend on the nature of the cells to be cultured. In 277 addition to culturing, there are temperature considerations when it comes to storing cells. The 278 maximum temperature for growth depends on the thermal sensitivity of secondary and tertiary 279 structures of proteins and nucleic acids. The minimum temperature depends mainly on the 280 freezing temperature. Different physiological groups of microorganisms adapt to different 281 temperature. Psychrophiles have optimal temperatures for growth below 15°C. Mesophilic have 282 optimal growth temperatures in the range between 20 and 40°C. Thermophiles grow best 283 between 50 and 70°C. There are known thermos-extremophiles growing at temperature higher 284 than 70°C [XX-1]. For example, prokaryote cells E. coli's optimal temperature to live in is 285 around 37°C and Streptomyces griseus is at 25 to 35C; for eukaryote cell Saccharomyces 286 cerevisiae is 31 to 35°C and Saccharomyces uvarum around 30 to 36°C [XX-2].Considering a 287 temperature increase limited to 4°C (compatible with cell culture), restricted operating conditions 288 (minimum flowrate and maximum mixing rate) can be estimated from Eq. (13 and 14) including 289 thermal balance and power consumption.

290

(figure 3a and b)

291 3.2 Analytical studies

Analytical results will be discussed concerning distribution and cumulative distribution functions, and moments. Two flow regimes (laminar regime $Re_{mixing} < 1 \times 10^3$, turbulent $Re_{mixing} > 3 \times 10^4$), different Q_f and N (laminar regime: $Q_f = 25$, 50, 100 L/h, N=0, 2, 10, 25 Hz; turbulent regime: $Q_f = 50$, 100, 150, 300 L/h, N=0, 2, 10, 25, 50 Hz) were scrutinized in agreement with established friction and power consumption curves, and corresponding to industrial practice. 298 *3.2.1 Distribution and cumulative distribution functions*

Following Thereska et al. [25], experimental inlet and outlet RTD signals, x(t) and y(t) are formulated. The measured conductivity values were converted into concentration values at a reference temperature (25°C). The concentration profiles were obtained as a function of time and figure 4 illustrates typical inlet and outlet RTD normalized signals, in laminar and turbulent regimes respectively.

304

(figure 4a and b)

305 *Qualitative comparison on inlet and outlet RTD signals and mass balance*

306 Inlet signal, x(t) is only affected by nominal Q_f (from 25 up to 300L/h in laminar and 307 turbulent regimes). Inlet curve, x(t) at given operating conditions indicates a good repeatability. Considering normalized inlet signal, $\tau \cdot x(t) = f(t/\tau)$ almost identical functions were observed 308 309 in laminar and turbulent regimes which indicate an accurate reproducibility (control of injected 310 volume and tracer amount, injection time and repetition). However, x(t) peak values in laminar 311 regime are significantly inferior to turbulent regime whereas spreading is larger. Differences can 312 be attributed to injection device (efficiency of static mixer), injection time and flow regime. In 313 turbulent regime, tracer solution was well-mixed and homogenized in bulk before passing 314 through the measurement cell, then detection was accurate. In laminar regime, tracer solutions 315 were poorly mixed in laminar regime, with inhomogeneous concentration along a cross-section, 316 then detection might present slight deviation between experiments.

Outlet signals, y(t) are affected by N and Q_f and express the RVF behaviour. Figures 3a and 3b illustrate the impact of N on outlet functions. In laminar regime, y(t) at 0 Hz appears as a sharp and unsmoothed signal. Under these conditions, RVF module exhibits none mixing efficiency and our remarks are similar to inlet signal. For N>0Hz, y(t) present a stable outlet 321 signal with a lower peak and consequently a larger spreading. In turbulent regime, smooth 322 signals were obtained due to turbulence, internal geometrical complexity and mixing rate. The 323 increase in mixing rate generates a larger asymmetric Gaussian curve.

324 Impact of mixing rate N and feeding flowrate Q_f in turbulent and laminar regimes

Figures 5a and 5b compare the outlet RTD signals, y(t) as a function of *N* and regime for a given $Q_f(100 \text{ L/h})$.

327

(figure 5a and b)

In turbulent regime, it shows the sliding of peak value from 0.037 to 0.021 1/s, with the Nincrease from 0 to 25 Hz. In laminar regime, peak value slides from 0.021 to 0.014 1/s with the Nincrease from 0 to 50 Hz.

331 Cumulative distribution functions, Y(t) were plotted in figures 6a and 6b as a function of N and Q_f . With various Q_f , it can be clearly seen in both flow regimes, Y(t) of higher Q_f has steeper 332 333 acceleration than the lower Q_f as expect, consider that the molecule flow out from the RVF 334 module faster when it was driven by a higher Q_f . On the other hand, with various N, Y(t) of 335 turbulent regime(see figure 5b) has a noticeable growth: in each cluster of colored curves, with 336 the increase of N, the Y(t) turn to be flatter, and slowly reach the plateau of the curve. Yet there is 337 also a large difference of N in laminar regime (see figure 5a), but it has no such significant 338 impact.

339

(figure 6a and b)

340 *3.2.2 Discussion of moments*

341 The average operating conditions and calculated RTD parameters under laminar and 342 turbulent regimes are reported in table 1 and table 2. In some extreme operating conditions, 343 significant temperature increase (difference between inlet and outlet) was observed both in laminar and turbulent flow. For example, temperature increased by 10.8 °C and 8.8 °C 344 345 respectively in laminar ($Q_f=25$ L/h, N=25 Hz) and turbulent regimes ($Q_f=50$ L/h, N=50 Hz). 346 (table 1) 347 (table 2) *Moment of 1st order (mean residence time)* 348 349 Moment of first order Γ^1 represents the mean residence time t_s in the system, as expressed by Eq. (5). The product $t_s \cdot Q_f$ represents the effective volume V_l , indicates the used volume in 350 351 the system. Figures 7a and 7b report the evolution of mean residence time, t_s as a function of mixing 352 353 rate, N in laminar and turbulent regimes. 354 (figure 7a and b) In laminar regime, t_s remain constant at a given Q_f , with a mean value $t_s=50\pm5$, 95±3 and 355 356 152±2 s for 25, 50 and 100 L/h respectively, whatever the N is. In turbulent regime, t_s increases linearly as a function of N at a given Q_f , t_s can be generally described by Q_f and N: 357 $t_s = \alpha N + \tau_0$ (15) V_1 can be expressed by: 358 $V_1 = t_s \cdot Q_f = \alpha N + V_0 = 0.0106 \cdot N + 1.15$ (16)Where α and τ_0 is as function of $1/Q_f (R^2 \ge 99\%)$, and $t_s = \frac{V_1}{Q_f}$ and $\tau_0 = \frac{V_0}{Q_f}$. 359

Figure 8 plots V_I as a function of mixing Reynolds number. In laminar flow which in the range of Remixing<×10³, V_I is constant at the given Q_f , it shows V_I = 1.12 L ± 0.06 at Q_f = 25L/h, V_I =1.27 L ± 0.07 at Q_f =50L/h, and V_I =1.34 L ± 0.02 at Q_f =100 L/h. V_I is dependent from Q_f , and *N* does not seem to have significant impact on V_I . In turbulent regime, initial used volume (*N*=0 Hz) is at about 1.15 L and it extends with the increase of RVF mixing rate. In this case, the effective volume is only mixing rate dependent whereas flowrate does not have any effect. It is noticeable that V_I and t_s depend on mixing rate in turbulent regime.

367

(figure 8)

368 V_l and t_s reflect the real movement of the tracers in RVF module (including shortcut, bypass 369 or dead-zone that may exist). While τ represents the holding time based on the assumption that 370 tracer molecules ideally move all over RVF module (V_{RVF}). Therefore, effective ratio defined by 371 t_s/τ or V_l/V_{RVF} becomes one of the important parameters to diagnose the flow system. The closer 372 is the ratio to 100%, the closer the system to the perfect mixer. In laminar regime, the effective 373 ratio was equal to 70%, 79%, and 84% at 25, 50, 100 L/h respectively. The main factor that 374 improves this ratio is the increase of the Q_{f} , and it seems mixing rate doesn't drive the fluid to 375 reach all over the system. To explain, at first, N was limited to 25 Hz which might be not high 376 enough to drive the fluid. Besides, it probably due to that laminar regime is very stable as widely 377 known, the mixing effect in this kind of flow regime is poor. However, under turbulent regime, 378 initial effective volume equal to 1.15 L (N=0 Hz) is almost the same value as under laminar 379 regime at 25 L/h, and it has a rapid growth as the increase of mixing rate. One can note that V_l is 380 tending towards V_{RVF} which is corresponding to an optimal use of the available volume. Overall, 381 these observations lead to the fact that, under laminar regime, mixing is mainly governed by the flowrate Q_f ; while under turbulent regime, mixing is largely governed by the stirring speed N. 382

383 *Centred moment of 2nd order, (reduced variances,* β^2 *)*

Figure 9 plots the evolution of the experimental reduced variance β^2 against mixing Reynolds. There is a significant decrease in reduced variance under both laminar and turbulent regimes. It is noticeable that the higher Q_f was, the closer to a perfect mixer, the system seems, whereas with higher Re_{mixing} it tends to a plug flow reactor. Considering $Q_f = 50$ and 100 L/h in both flow regime, two logarithmic regressions are plotted, which denotes that β^2 and mixing effect (Re_{mixing}) are somehow related.

390

(figure 9)

391 *3.23 Reduced signal of outlet distribution function y*(*t*)

In laminar regime, effective volume was highly correlated to Q_f whereas in turbulent regime, N played a role as the dominant parameters. In order to confirm or to refute the uniqueness of hydrodynamics behaviour of RVF module, distribution functions y(t) were reduced by experimental mean residence time t_s and expressed as $y(t/t_s) \cdot t_s$.

Figure 10 plots these reduced distributions as a function of t/t_s at the given operating condition. Then overlapping coefficient, $OVL = \int_0^t \min[f_1(x), f_2(x)]dx$) is calculated to compared experimental data.

In laminar regime at given feeding flowrate $Q_f = 100$ L/h, (see figure 9a1) reduced distribution functions are separated and the peak values slide from 1.83 to 1.07 with the increase of *N*. Furthermore, for three feed flowrates, Q_f at given *N* (see figure 9a2), reduced distribution functions are perfectly overlapped (OVL= 0.86).

403 Figure 10a1 and a2 indicate that RVF behaviour is the same at a given N whatever Q_f 404 values.

In turbulent regime, reduced distribution functions, $y(t/t_s) \cdot t_s$ are perfectly overlapped (Figure 10a, with OVL=0.85) in all conditions indicating the uniqueness of hydrodynamics behaviours. It can be reasonably assumed that flow behaviour in turbulent regime is somehow the same, whatever flowrate, Q_f and mixing rates, N are. Since the reduced distribution functions showed its uniqueness in whole or in part in different flow regime, flow behaviours in the RVF module probably can be explained by a unique model with an equivalent known system.

411

(figure 10a and b)

412 3.3 Systemic analysis and modelling of RTD

413 After the analytical studies of RTD, the hydrodynamic behaviours were described by a 414 systemic analysis by modelling RTD outlet signals using DTS Progepi v4.2. Our objective is to 415 model and to establish an analytical solution of RTD functions considering the complexity of 416 RVF module.

417 3.31 Proposal of reactor models

Three models were proposed to fit the outlet function y(t) of RVF module in order to obtain a better agreement. Inlet function x(t) was taken into account by considering the convolution with outlet function y(t). For each tested configuration, a Plug Flow reactor was used to describe the tubes upstream and downstream of the RVF module. The RVF module was described by three different associations of ideal reactors (figure 11).

423

(figure 11)

424 Model 1: A plug flow reactor in serie with a plug flow reactor with axial diffusion. The 425 value of the volume was fixed equal to V_{RVF} and adjusting Peclet number Pe_L . Consequently, this 426 model exhibit only 1 degree of freedom (Pe_L). RTD function is described by an analytical 427 solution (Eq.17).

$$E(t) = \frac{1}{2} \left(\frac{Pe_L}{\pi\tau t}\right)^{1/2} exp\left(-\frac{Pe_L(\tau-t)^2}{4\tau t}\right), where \ \tau = \frac{V_{RVF}}{Q_f}$$
(17)

with
$$\frac{t_s}{\tau} = 1 + \frac{2}{Pe_L}, \quad \beta^2 = \frac{\sigma^2}{t_s^2} = \frac{2}{Pe_L} + \frac{8}{Pe_L^2}$$
 (18)

428 Model 2: A plug flow reactor in serie with J continuous stirred tank reactors. The value of 429 the volume was fixed equal to V_{RVF} . This second model is defined by the value of *J*, the 430 residence time of the plug reactor (τ_p) and the residence time of the J CSTR (τ_{cs}). This model 431 has been tested in previous studies [17, 26-28]. The corresponding expressions for the transfer 432 function, *G*(*s*) and *E*(*t*) are formulated by Eq. (19) and Eq.(20):

$$G(s) = \frac{exp(-s\tau_p)}{\left(1 + \left(\frac{s\tau_{cs}}{J}\right)\right)^{J}}, where \ \tau = \frac{V_{RVF}}{Q_f}$$
(19)

$$E(t) = H(t - \tau_p) \left(\frac{J}{\tau_{cs}}\right)^J (t - \tau_p) exp\left(\frac{-J(t - \tau_p)}{\tau_{cs}}\right)$$
(20)

433 Model 3: A plug flow reactor in serie with J continuous stirred tank reactors. This model is 434 a modified version of model 2. Indeed, the value of the volume was taken equal to V_1 435 (experimental values from Tables 1 and 2) instead of the total volume of RVF module.

436	3.3.2	Model	l ad	justment and	l comparison
				/	

437	Even if inlet signals can be neglected, we observed that the injection were slightly different
438	from ideal Dirac functions, especially in laminar regime. In order to better fit the experimental
439	functions, $y(t)$, the inlet functions $x(t)$ were considered with all models to simulate $E(t)$.
440	Figure 12 plots the modelling curve $y(t)'$ of models 1, 2 and 3 fitting with the experimental data
441	y(t) (one operating condition).
442	(figure 12)
443	Table 3 sums up overlapping coefficient values for models 1, 2 and 3.
444	(table 3)
445	Significantly, model 3 can be preferred in both laminar and turbulent regimes. Table 4 shows the
446	values of <i>J</i> for this model.
447	(table 4)
448	To conclude this systemic approach, the model 3 defined by the effective volume V_1 and
449	J= 2.5 ± 0.6 can be chosen to describe flow behaviour in RVF module.
450	

451 **4 Conclusions**

For MBR using dynamic filtration module, RTD is an efficient way to diagnose bioprocess efficiency. In present work, tracer experiments were applied to investigate RTD in an impellerrotating filter (RVF module) in laminar and turbulent regimes.

Firstly, experimental observation and analysis of RTD in this complex module are discussed. It has allowed the characterization of the flow behaviour and the determination of defined parameters.

458 Results mainly demonstrate that:

459 • Thermal effect was introduced in order to have the knowledge of temperature change and 460 heat dissipation in a bioprocess context (cell culture). Thermal balance (heat dissipation) is 461 correlated to mixing power (power consumption curve). The temperature increased dramatically 462 at high rotation speed, especially in laminar flow (ex. +11°C under operating conditions Qf=25 463 L/h and N=25 Hz). In turbulent flow, with Qf =50 L/h and N=50 Hz, temperature increased up to 464 9°C. Considering a temperature increase limited to 4°C (compatible with cell culture), restricted 465 operating conditions (minimum flowrate and maximum mixing rate) can be estimated from 466 thermal balance and power consumption curve.

• Under laminar regime, mean residence time, ts and effective volume, V1 remained constant for all imposed flowrates Q_f , whatever mixing rate, N. Under turbulent regime, t_s increased linearly as a function of N at a given Q_f . The effective volume V_I was only N dependent, whereas Q_f had no effect. V_I ranged from 1.15 up to 1.6 L corresponding to effective ratio from 72% up to 100%. In brief, the governing parameter of the mixing in laminar regime was mainly the feeding flowrate Q_f , while in turbulent regime it was the mixing rate, N. 473 • Centred moments of 2^{nd} order, reduced variances β^2 , are as a linear function of mixing 474 Reynolds number for the given flowrate, β^2 slightly increased with Q_f .

Secondly, a systemic analysis lead to model of RTD. Three conventional reactor models (exhibiting analytical solutions) were evaluated to predict the distribution function, E(t)considering convolution of inlet, x(t) and outlet, y(t) functions. Modelling of RTD by PFR+(J) CSTR model (effective volume, V_1 and $J = 2.5\pm0.6$) accurately estimate the mean residence time and its associated distribution.

Interactions between biological matrices and hydrodynamics in Dynamic Filtration must be deeply studied to intensify bioprocesses. Even if RTD lead to identify critical operating conditions, global bioprocess performances arise from a local interactions including velocity distribution, shear stress, turbulence, local temperature, etc. [17]. Future studies might include:

The investigation of local hydrodynamic (velocity fields, local velocity profiles, wall
shear stress etc.) through PIV (Particle Image velocimetry) measurement [19, 28, 29] and CFD
approach (fluid streamlines) in order to quantify local shear stress and its time-evolution as well
as the flow pattern and coherent flow structures;

488 • The investigation of RTD with Non-Newtonian fluids to understand and simulate the
489 rheological behaviour of biological suspensions;

490

491

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- 610

612 Figures caption

- Fig. 1 Scheme, top view of one filtration cell and global view of Rotating and VibratingFiltration module.
- Fig. 2 Experimental setup and injection device (a) schematic of experimental setup, C1, C2:
 Electrical conductivity sensors; (b) Injection device; (c) Photo of experimental setup.

- 620 Fig. 4 Inlet and outlet distribution functions, x(t) and y(t) in (a) laminar regime, operating
- 621 conditions: BREOX 35% solution, μ =0.35 Pa·s, Q_f=100 L/h, N=0 (T_{in}=22.7 ± 0.01°C,
- 622 $T_{out}=23.0 \pm 0.02^{\circ}C$) and 25 Hz ($T_{in}=22.2 \pm 0.14^{\circ}C$, $T_{out}=26.3 \pm 0.13^{\circ}C$); and in (b) turbulent
- ferror regime, operating conditions: water, $\mu=1$ mPa·s, $Q_f=100$ L/h, N=0(T_{in}=13.0 ± 0.12°C,

624
$$T_{out}=13.1 \pm 0.11^{\circ}C$$
 and 50 Hz ($T_{in}=14.8 \pm 0.02^{\circ}C$, $T_{out}=19.3 \pm 0.02^{\circ}C$).

- Fig. 5 Outlet distribution functions, y(t) in (a) laminar regime, operating conditions: μ =0.35 Pa·s,
- 626 $Q_f=100$ L/h, N=0, 2, 10, 25 Hz; and in (b) turbulent regime, operating conditions: water, 627 $\mu=1$ mPa·s, Q=100 L/h, N=0, 2, 10, 25, 50 Hz.
- Fig. 6 Cumulative functions, Y(t) in (a) laminar regime, operating condition: 35% BREOX solution μ =0.35 Pa·s, Q_f=25, 100 L/h, N= 2, 10, 25 Hz; and for (b) turbulent regime, operating conditions: water, μ =1 mPa·s, Qf=50, 150, 300 L/h, N=2, 10, 25, 50 Hz.
- Fig. 7 Mean residence time, t_s as a function of mixing rate, N in (a) laminar regime, operating conditions: BREOX 35% solution μ =0.35 Pa·s, Q=25, 50, 100 L/h, N=0, 2, 10, 25 Hz; and

633 for (b) turbulent regime, operating conditions: water, μ=1 mPa·s, Q=50, 100, 150, 300 L/h,
634 N=0, 2, 10, 25, 50 Hz.

Fig. 8 Efficient volume V_1 as a function of mixing Reynolds number, Re_{Mixing}. In laminar regime, operating conditions: BREOX 35% solution μ =0.35 Pa·s, Q_f=25, 50, 100 L/h, N=2, 10, 25 Hz; in turbulent regime, operating conditions: water, μ =1 mPa·s, Q_f=50, 100, 150,

638 300 L/h, N= 2, 10, 25, 50 Hz.

639 Fig. 9 Reduced variance, $β^2$ as a function of mixing Reynolds number Re_{Mixing}. In laminar 640 regime, operating conditions: BREOX 35% solution μ=0.35 Pa·s, Q=25, 50, 100 L/h, N=0,

641 2, 10, 25 Hz; un turbulent regime, operating conditions: water, Q=50, 100, 150, 300 L/h, N=
642 2, 10, 25, 50 Hz.

643 Fig. 10 Reduced distributiuion function, $t_s.y(t)$ as a function of reduced time, t/t_s , in (a) laminar

regime (operating conditions in a1: BREOX 35% solution, μ =0.35 Pa·s, Q=100 L/h, N=0, 2,

645 10, 25 Hz, in a2: BREOX 35% solution, μ=0.35 Pa·s, Q=25, 50, 100 L/h, N=25 Hz) and in

646 (b) turbulent regime (operating conditions: water, μ =1 mPa·s, Q=50, 300 L/h, N=0, 50 Hz).

Fig. 11 Proposed structure to describe RTD within RVF module; Model 1: Plug flow reactor open to diffusion by imposing V_{RVF} ; Model 2: perfect mixing cell in series by imposing V_{RVF} and model 3: perfect mixing cell in series by imposing the efficient volume, $V_{1.}$

Fig. 12 Comparison of the fitting curves with model 1, 2 and 3. (a): laminar regime with BREOX 35% solution, Q_f =100 L/h, N=25 Hz; (b) turbulent regime with water, Q_f =100 L/h, N=10 Hz.

654

655 List of tables:

656 Table 1: Operating conditions and calculated RTD parameters in laminar regime (SD: standard

deviation).

- Table 2: Operating conditions and calculated RTD parameters in turbulent regime (SD: standarddeviation).
- 660 Table 3: Overlapping coefficient values for models 1, 2 and 3.
- Table 4 : J value in Model 3

662