FLOW INVESTIGATION IN AN INNOVATING DYNAMIC FILTRATION MODULE USING TRACING METHODS

Xiaomin XIE[a,e], Christophe ANDRE[b,c,d *], Nicolas DIETRICH[a,e], Philippe SCHMITZ[a,e], Luc FILLAUDEAU[a,e]

[a] LISBP, Université de Toulouse, CNRS UMR5504, INRA UMR792, INSA, Toulouse, France
[b] UC Lille, HEI, Laboratoire de Génie des procédés, 59046 Lille, France
[c] INRA, UR638, PIHM, Villeneuve d’Ascq, France
[d] UMET, CNRS-UMR8207, Université de Lille 1, Villeneuve d’Ascq, France
[e] FERMAT, INP Toulouse, CNRS, INSA Toulouse, UPS, France.

[*]corresponding author:
UC Lille, HEI, Laboratoire de Génie des procédés, 59046 Lille, France
INRA, UR 638, PIHM, BP 20039,369 rue Jules Guesde, 59651 Villeneuve d’Ascq, France
E-mail: christophe.andre@yncrea.fr

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.

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

Keywords:

Dynamic filtration; thermal balance, Residence time distribution; modelling; systemic approach.

Nomenclature:

Latin Letters		
C	Concentration (salt)	[g/L]
C_p	Specific heat capacity	[J/ (kg·°C)]
d_m	Impeller diameter	[m]
E_a	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]
T	Temperature	[°C]

TMP	Transmembrane pressure	[bar]
V_1, V_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 2nd 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	

1 Introduction

The principle of Dynamic Filtration (DF) is to generate complex hydrodynamic perturbations (magnitude of velocity and shear stress, time dependent) by mechanical movement (rotation, vibration, oscillation) of membrane or external mechanical forces close to the membrane, to better control membrane fouling. The external force applied to the system can be longitudinal, transverse, torsional, or with a mix of these motions. In the recent decades, many efforts have been done to study hydrodynamics and to develop novel DF modules at lab scale or pilot scale. In each device, the controls of local and global performances (permeability, fouling, limitations) are closely related to our knowledge of local and global hydrodynamic. The existing DF modules can be classified in terms of mechanical movement as cylindrical rotating filter (such as *Biodruck-filter*[1], *Biopurification System*[2], *RDF filter*[3]), rotating flat membrane filter (such as *CRD filter*[4], *MSD filter*[5], *RDM module*[6-8], *DYNO filter*[9], *RVF module*[10-12]), and vibrating filter (such as *VSEP filter*[13], *VMF filter*[14] *and hollow fibber filter*[15,16]). Compare with dead-end (DE) and cross-flow (CF) filtration, DF has been proved (1) by reducing environmental impact (low loop volume), (2) by reducing energy consumption, uncoupling between the conventional driving force (from feeding flowrate) and wall shear stress, working under low transmembrane pressure (TMP). Enhancements in DF are mainly attributed to complex hydrodynamics near the membrane surface due to the various motions (rotation, 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 the membrane surface always leads to a decline of permeate flux over time. Many efforts have been done to recover or maintain a high efficiency and quality of permeate by using plenty of low viscosity feeding fluid. However, less attention has been paid to the fluid transport and flow 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 information, Residence Time Distribution (RTD) is a crucial approach to diagnose the flow performance associated with degree of mixing and shearing which play an important role in the final product quality[17].

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

2 Materials and methods

2.1 Filtration module

The DF module, shown in fig. 1, is called Rotating and Vibrating Filtration (RVF technology – patent no. FR-97-14825) [18]. It is designed for biological or food liquids. RVF 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), and in each cell, a three-blade impeller (flat blade, d_m = 138 mm, thickness = 8 mm) is driven by a central shaft continuously rotating (up to 50 Hz) in a 14 mm gap between two porous flat support (membrane support) which drains the permeate. It gives a 3 mm gap between the impeller and the membrane surface. This simple mechanical device runs continuously and generates a high shear rate as well as a hydrodynamic perturbation in the small gap, TMP (up to 300 kPa) and rotation frequency can be adjusted to optimize the operating conditions.

(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: $Re_{mixing}<1\times10^3$; transition regime: $1\times10^3<Re_{mixing}<3\times10^4$; and turbulent regime: $Re_{mixing}>3\times10^4$.

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 µS/cm, precision: ±1%/decade and KEMOTRON type 9147 Integral flow, range: 5-2000000 µS/cm, precision: ±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.

(figure 2)

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

2.3 Experimental fluids and tracers

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

The RTD experiments were restricted to two Newtonian fluids (BREOX solution and water) for laminar and turbulent regime respectively. A water soluble and viscous transparent Newtonian fluid (BREOX® Polyalkylene Glycol 75 W 55000, BASF) was used to achieve laminar regime, and water was used for the turbulent regime. For BREOX solution, thermo-physical properties including viscosity μ [Pa·s], density ρ [kg/m^3], heat capacity C_p [J/(kg·°C)] 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 density meter (DE40, SN: MPK38384, Mettler Toledo, France, range: 10^{-4} to 3 g/cm^3 ± 10^{-4} g/cm^3, 4 to 90°C ± 0.05°C), and a differential scanning calorimeter (Micro DSC-III, SN: 60/50287.06.102, Setaram, France, temperature range: -20 to +120°C, thermal kinetics: 10^{-3} to +1.2°C and fluxmeter range: 0.2 µW 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 45°C) as follows:

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

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

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

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, $T_r = 20°C$. 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 (with μ = 0.35 Pa·s, Cp = 3650 J/(kg·°C) and ρ = 1040 kg/m^3 at T = 20°C). Temperature of the feeding tank was controlled at about 20°C by adjusting the thermostat (room temperature was 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 $\rho = 998.2$ kg/m^3, specific heat capacity C_p = 4181.8 J/(kg·°C) at constant pressure, viscosity μ=1.0 mPa·s. (under 100 kPa).

2.4 Methodology

2.4.1 Theory of Residence Time Distribution (RTD)

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

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

and

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

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

$$E(t)=\frac{C_{out}(t)}{\int_0^\infty C_{out}(t)dt} and\, F(t)=\int_0^\infty E(t)dt \qquad \text{(6) and (7)}$$

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

$$\Gamma^1=t_s=\int_0^\infty tE(t)dtand\,\tau=\frac{V}{Q_f} \qquad \text{(8) and (9)}$$

and

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

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].

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

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.

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 raw data 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 temperature T_{in} and T_{out}, were recalculated at T_{ref} (25°C), based on equation $\sigma = \sigma_{25°C} + a \cdot T^{n}_{25°C} \cdot (T - 25)$, with *a=0.022* and *n=0.93* [26]. Base line was then subtracted and conductivity signal $\sigma - \sigma_0(t)$ was converted into salt concentration, *C*. Mass balance was established by comparing inlet and outlet signals, $q_{in} = \int_0^{\infty} C_{in}(t) \cdot Q(t) \cdot dt$ and $q_{out} = \int_0^{\infty} C_{out}(t) \cdot Q(t) \cdot dt$. 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 $y(t) = \frac{C_{out}(t)}{\int_0^{\infty} C_{out}(t)dt}$, by taking the contribution of injection effect into account; Thereafter, 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 perfect inlet impulsion, x(t) is a Dirac distribution and distribution function E(t) will be equal to y(t). In the fourth step, moment and centred moments were discussed concerning mean residence time, and RTD curve including dispersion, asymmetry, and spreading. These moments enable to diagnose the behaviour of the reactor as a function of flow and mixing regimes.

In the fourth step, a systemic approach is then conducted by software (DTS Progepi, RTD Software Analysis). This software was used to correctly modelling the experimental response to an input of any complex set-up consisted of connected elementary reactors [27]. This step is based on the identification of parameters (hypothesis of a model based on the interconnection of elementary reactors more or less complex) by adjusting the simulated response according to the experimental data. The software realizes optimization by Rosenbrock's method, allowing 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 model and the experimental curve *y(t)*.

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.

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:

$$\Delta \mathrm{P} \cdot \mathrm{Q} + \iint_{A} \sum_{j=1}^{3} \sum_{i=1}^{3} \tau_{if} n_i U_j \, ds + \iint_{A} \sum_{i=1}^{3} (-P) n_i U_i \, ds = \iiint_{D} \rho \varepsilon_V dv \qquad (13)$$

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

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

$$P_{eff} = \iiint_{D} \rho \varepsilon_V dv = \rho C_p (T_{inlet} - T_{outlet}) Q_f \qquad (14)$$

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 flow-rate. 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], $\mathrm{Np} = \left(\mathrm{B}^{\frac{1}{n}} + \left(\frac{\mathrm{Kp}}{\mathrm{Re}_{\mathrm{mixing}}}\right)^{\frac{1}{n}}\right)^{n} \cong \frac{\mathrm{Kp}}{\mathrm{Re}_{\mathrm{mixing}}}$, (equation (14)) where n=2, B=0.10, Kp = 520, by knowing mixing Reynolds.

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

(figure 3a and b)

3.2 Analytical studies

Analytical results will be discussed concerning distribution and cumulative distribution functions, and moments. Two flow regimes (laminar regime $Re_{mixing}<1\times10^{3}$, turbulent $Re_{mixing}>3\times10^{4}$), 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.

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.

(figure 4a and b)

Qualitative comparison on inlet and outlet RTD signals and mass balance

Inlet signal, *x(t)* is only affected by nominal Q_f (from 25 up to 300L/h in laminar and turbulent regimes). Inlet curve, *x(t)* at given operating conditions indicates a good repeatability. Considering normalized inlet signal, $\tau \cdot x(t) = f\left({}^{t}/_{\tau}\right)$ almost identical functions were observed in laminar and turbulent regimes which indicate an accurate reproducibility (control of injected volume and tracer amount, injection time and repetition). However, *x(t)* peak values in laminar regime are significantly inferior to turbulent regime whereas spreading is larger. Differences can be attributed to injection device (efficiency of static mixer), injection time and flow regime. In turbulent regime, tracer solution was well-mixed and homogenized in bulk before passing through the measurement cell, then detection was accurate. In laminar regime, tracer solutions were poorly mixed in laminar regime, with inhomogeneous concentration along a cross-section, 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 signal with a lower peak and consequently a larger spreading. In turbulent regime, smooth signals were obtained due to turbulence, internal geometrical complexity and mixing rate. The increase in mixing rate generates a larger asymmetric Gaussian curve.

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 L/h).

(figure 5a and b)

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

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 acceleration than the lower Q_f as expect, consider that the molecule flow out from the RVF module faster when it was driven by a higher Q_f. On the other hand, with various *N*, *Y(t)* of turbulent regime(see figure 5b) has a noticeable growth: in each cluster of colored curves, with the increase of *N*, the *Y(t)* turn to be flatter, and slowly reach the plateau of the curve. Yet there is also a large difference of *N* in laminar regime (see figure 5a), but it has no such significant impact.

(figure 6a and b)

3.2.2 Discussion of moments

The average operating conditions and calculated RTD parameters under laminar and turbulent regimes are reported in table 1 and table 2. In some extreme operating conditions, 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 respectively in laminar (Q_f= 25 L/h, N=25 Hz) and turbulent regimes (Q_f= 50 L/h, N=50 Hz).

(table 1)

(table 2)

Moment of 1st order (mean residence time)

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_1, indicates the used volume in the system.

Figures 7a and 7b report the evolution of mean residence time, t_s as a function of mixing rate, N in laminar and turbulent regimes.

(figure 7a and b)

In laminar regime, t_s remain constant at a given Q_f, with a mean value t_s=50±5, 95±3 and 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:

$$t_s = \alpha N + \tau_0 \quad (15)$$

V_1 can be expressed by:

$$V_1 = t_s \cdot Q_f = \alpha N + V_0 = 0.0106 \cdot N + 1.15 \quad (16)$$

Where α and τ_0 is as function of $1/Q_f$ ($R^2 \geq 99\%$), and $t_s = \frac{V_1}{Q_f}$ and $\tau_0 = \frac{V_0}{Q_f}$.

Figure 8 plots V_1 as a function of mixing Reynolds number. In laminar flow which in the range of Remixing$<\times 10^3$, V_1 is constant at the given Q_f, it shows V_1= 1.12 L ± 0.06 at Q_f= 25L/h, V_1=1.27 L ± 0.07 at Q_f =50L/h, and V_1=1.34 L ± 0.02 at Q_f =100 L/h. V_1 is dependent from Q_f, and N does not seem to have significant impact on V_1. 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_1 and t_s depend on mixing rate in turbulent regime.

(figure 8)

V_1 and t_s reflect the real movement of the tracers in RVF module (including shortcut, bypass or dead-zone that may exist). While τ represents the holding time based on the assumption that tracer molecules ideally move all over RVF module ($\mathrm{V_{RVF}}$). Therefore, effective ratio defined by $t_s/\ \tau$ or V_1/V_{RVF} becomes one of the important parameters to diagnose the flow system. The closer is the ratio to 100%, the closer the system to the perfect mixer. In laminar regime, the effective ratio was equal to 70%, 79%, and 84% at 25, 50, 100 L/h respectively. The main factor that improves this ratio is the increase of the Q_f, and it seems mixing rate doesn't drive the fluid to reach all over the system. To explain, at first, N was limited to 25 Hz which might be not high enough to drive the fluid. Besides, it probably due to that laminar regime is very stable as widely known, the mixing effect in this kind of flow regime is poor. However, under turbulent regime, initial effective volume equal to 1.15 L (N=0 Hz) is almost the same value as under laminar regime at 25 L/h, and it has a rapid growth as the increase of mixing rate. One can note that V_1 is tending towards V_{RVF} which is corresponding to an optimal use of the available volume. Overall, 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.

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.

(figure 9)

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, $OVL = \int_0^t \min\left[f_1(x), f_2(x)\right] dx$) 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).

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

(figure 10a and b)

3.3 Systemic analysis and modelling of RTD

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

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).

(figure 11)

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

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

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

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

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

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

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

3.3.2 Model adjustment and comparison

Even if inlet signals can be neglected, we observed that the injection were slightly different from ideal Dirac functions, especially in laminar regime. In order to better fit the experimental functions, *y(t), the* inlet functions *x(t)* were considered with all models to simulate *E(t)*.

Figure 12 plots the modelling curve $y(t)^{'}$ of models 1, 2 and 3 fitting with the experimental data *y(t)* (one operating condition).

(figure 12)

Table 3 sums up overlapping coefficient values for models 1, 2 and 3.

(table 3)

Significantly, model 3 can be preferred in both laminar and turbulent regimes. Table 4 shows the values of *J* for this model.

(table 4)

To conclude this systemic approach, the model 3 defined by the effective volume V_1 *and* J=2.5±0.6 can be chosen to describe flow behaviour in RVF module.

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 impeller-rotating 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.

Results mainly demonstrate that:

- Thermal effect was introduced in order to have the knowledge of temperature change and heat dissipation in a bioprocess context (cell culture). Thermal balance (heat dissipation) is correlated to mixing power (power consumption curve). The temperature increased dramatically at high rotation speed, especially in laminar flow (ex. +11°C under operating conditions Qf=25 L/h and N=25 Hz). In turbulent flow, with Qf =50 L/h and N=50 Hz, temperature increased up to 9°C. Considering a temperature increase limited to 4°C (compatible with cell culture), restricted operating conditions (minimum flowrate and maximum mixing rate) can be estimated from 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_1 was only N dependent, whereas Q_f had no effect. V_1 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.

- Centred moments of 2nd order, reduced variances β^2, are as a linear funtion of mixing 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±0.6) 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;
- The investigation of RTD with Non-Newtonian fluids to understand and simulate the rheological behaviour of biological suspensions;

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Figures caption

Fig. 1 Scheme, top view of one filtration cell and global view of Rotating and Vibrating Filtration 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.

Fig. 3 Temperature variation of the test fluid with different mixing rate (A) and and Heat dissipation [W] as a function of calculated mixing power consumption [W].*********** Change figure numbering after *********************

Fig. 4 Inlet and outlet distribution functions, x(t) and y(t) in (a) laminar regime, operating conditions: BREOX 35% solution, μ=0.35 Pa·s, Q_f=100 L/h, N=0 (T_{in}=22.7 ± 0.01°C, T_{out}=23.0 ± 0.02°C) and 25 Hz (T_{in}=22.2 ± 0.14°C, T_{out}=26.3 ± 0.13°C); and in (b) turbulent regime, operating conditions: water, μ=1 mPa·s, Q_f=100 L/h, N=0(T_{in}=13.0 ± 0.12°C, T_{out}=13.1 ± 0.11°C) and 50 Hz (T_{in}=14.8 ± 0.02°C, T_{out}=19.3 ± 0.02°C).

Fig. 5 Outlet distribution functions, y(t) in (a) laminar regime, operating conditions: μ=0.35 Pa·s, Q_f=100 L/h, N=0, 2, 10, 25 Hz; and in (b) turbulent regime, operating conditions: water, μ=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 for (b) turbulent regime, operating conditions: water, µ=1 mPa·s, Q=50, 100, 150, 300 L/h, 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, 300 L/h, N= 2, 10, 25, 50 Hz.

Fig. 9 Reduced variance, β^2 as a function of mixing Reynolds number Re_{Mixing}.. In laminar regime, operating conditions: BREOX 35% solution µ=0.35 Pa·s, Q=25, 50, 100 L/h, N=0, 2, 10, 25 Hz; un turbulent regime, operating conditions: water, Q=50, 100, 150, 300 L/h, N= 2, 10, 25, 50 Hz.

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

List of tables: