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## **An innovative pulsed column applied to solid-liquid contacting: the BPC column.**

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**Keywords:** solid-liquid contacting, pulsed column, pulsation, process intensification.

### **Abstract:**

The innovative process presented is a novel type of pulsed column (BPC column) used as a solid/liquid contactor. An evolution in the design of the internals and in the shape of the pulsation has been set up. The initial objectives were to achieve a reactor of greatly longer solid residence time, independently of the liquid phase. Phase contacting is carried out in a 1.5 m height column. Liquid and solid are flowing countercurrently. Solid is fed in at the bottom of the column and flows upwardly, whatever the density difference between both phases. The behaviour of solid and liquid phases was investigated through the analysis of residence time distributions. The working conditions range has been determined and it was found that an optimal tuning of the operating conditions (pulsation parameters, flows control...) could minimize the influence of the size and the density of particles and uncouple residence times of both phases. The column has been manufactured, in order to handle a large range of solid-liquid systems (solid treatment, purification or reaction, raw materials, floating or dense particles, polydispersed particles). On the basis of the model calculations and design considerations, preliminary data can be given on the technical and economical feasibility of the various applications quoted above. The final aim of this study is to prove that, for a fixed production rate and a product quality, the new column can satisfy the specifications required and even exceed them.

### **1. Introduction**

In classical solid-liquid pulsed columns, sinusoidal oscillations are imposed to enhance mixing and mass transfer between two phases contacted. Pulsed flow offers an interesting alternative means of external agitation in columns used as liquid-liquid or solid-liquid contactors. But, in any case of solid/liquid contact, it was noticed that this pulsation mode was not efficient enough to allow a controlled behaviour of the solid phase. The problem is particularly met for the treatment of raw plants or polydispersed populations with complex physical properties. Oscillations can also be dedicated to the displacement of fluid or solid phases, in order to have a better control of residence times. The field of applications is clearly broad. However, it's probably in processes with solid phase that such a concept will be useful, as flooding problems, granulometric dispersion or short residence times can be met [1, 2, 3, 4, 5]. Indeed, in counter current column, with high liquid flow rate, large particles are slowed (so their residence time increases), while smallest particles are driven by the liquid and then can form plug at the solid inlet position. On the contrary, with low liquid flow rate (lower enough not to drive the smallest particles), the solid residence time range can be very short.

The main ideas we developed was, in one hand, to use a system in which the solid can be stopped in compartments while the liquid continues to flow. This has been obtained by the use of simple mechanic valves on a porous media through which the liquid can flow. In the other hand we used a non sinusoidal pulsation which has been developed and studied in previous work for solid-liquid

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systems [5, 6]. This pulsation is used to enhance the mixing of the two phases and to open the compartments and transport the solid phase.

The objective of this study is to demonstrate the feasibility of the new kind of internals in a continuous contactor using a non-sinusoidal pulsation. After a description of the apparatus, some results concerning the solid residence time are presented, focusing on a viable experimental domain.

## 2. Pilot description

### 2.1. Internals choice and description

Two kinds of technologies have been evaluated in order to be able to stop the solid while the liquid continues to flow. The first one is the valve type technology; the second one is composed of a sphere guided by pins. We can consider four cases function of the direction of the solid flow (downward or upward) and function of the valve which can normally open or normally closed. Tab. 1 shows the four cases and their evaluation function of some qualitative criteria.



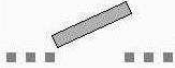


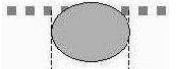

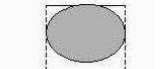
case	1	2	3	4
	Downward solid flow		Upward solid flow	
	Normally open	Normally closed	Normally closed	Normally open
Valve type technology				
Sphere type technology				
Technology simplicity	+	-	+	-
Solvent consumption	-	+	+	-
Particles segregation	-	+	+	-

Table 1: qualitative analysis of the technological choices

The “normally closed” position allows easy maintaining of the compartment closed for the solid. This is favourable to a normal consumption of solvent and particles segregation. At the contrary, the “normally open” position needs extra solvent flow to maintain the sealing of the compartments (i.e. to maintain the solid in the compartments). In that position, the particles dispersion due to the differences in sizes or density could be the same as a classical column.

The “valve type” technology has been tried but led to blocking of the moving element. The “sphere type” technology is then the one used in this work, even if it does not assure a complete sealing of the compartments to the solid.

Concerning the solid direction, a downward solid flow is easier to implement, but in order to demonstrate the feasibility of our pilot, we decided to use an upward solid flow. Indeed, if the pilot presents interesting operating domain and characteristic while working against the gravity, it will prove that it is possible to be free from the size and density difficulties (to a certain extent).

In conclusion of this preliminary study, we choose to work on the case 3 of Tab. 1, using a sphere. It means that the sphere is denser than the liquid and that the solid is flowing upwardly. Of course this study is neither complete nor quantitative, but it was a basis for the technological choices made.

## 2.2. Pilot description

The pilot BPC column is composed of a vertical cylindrical body which contains nine plates arranged and maintained by braces of constant length. Figure 1 shows a schematic presentation of such a column. The active part of the pilot column is 1 m high and 50 mm in diameter. It consists of plates made out of a filtering media (cloth or frit) (1a) and a valve composed of a free sphere guided by pins (1b) (Figure 2). This configuration allows avoiding any blocking of the mobile part during operation.

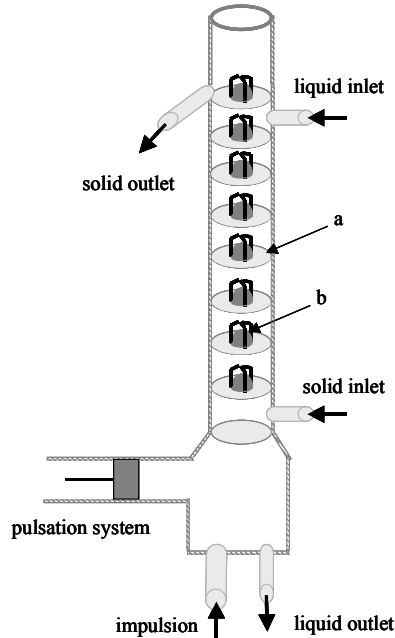


Figure 1 : pilot column

Figure 1: pilot column

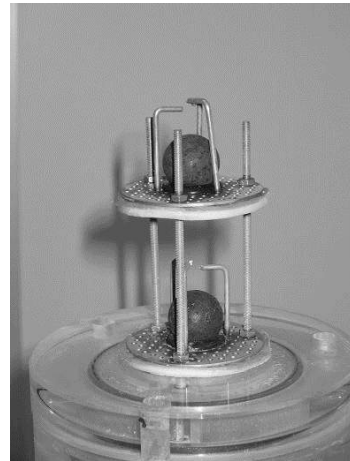


Figure 2 : pilot internal

## 2.3. Pulsation policy

The proposed system gives the opportunity to have a non-sinusoidal form of pulsation. The movement of the liquid is separated into two steps (Figure 3):

- A pulsation step: a classic sinusoidal pulsation is used to mix the two phases in the active part of the column. During this step, the sphere is not supposed to raise itself under the effect of the fluid. The solid is captured between two plates while the fluid can still circulate through the filtering medium. This pulsation is characterized by its frequency and amplitude  $f$  and  $A$ . This step lasts for a time equal to  $T_m$ ;
- An impulsion step: this second step allows to lift the spheres and then to force the solid to circulate from one tray to another. This impulsion is characterised by its frequency  $f_i$  and the volumic liquid flowrate  $Q_{imp}$ . This step lasts for a time equal to  $T_{imp}$ .

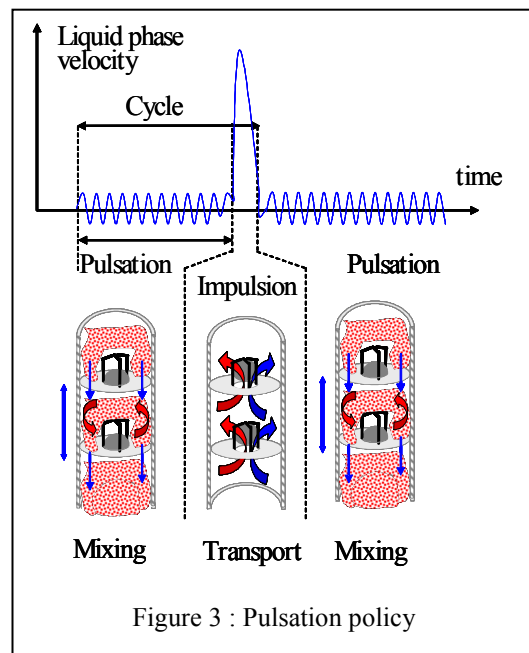


Figure 3 : Pulsation policy

The aperture of the plates and the spheres are 18 and 21 mm in diameter respectively. Liquid and solid are flowing countercurrently, the pulsation is ensured by a mechanical system and the impulsion by a liquid pump. The tested solid was composed of 900 and 200  $\mu\text{m}$  polystyrene spheres (density equal to 1.05). The volumic liquid flowrate at the top of the column is written  $Q_L$ , while  $Q_S$  is the solid flowrate at the bottom of the column. Each of the  $N_{\text{comp}}$  compartment is characterized by its height and its volume ( $H_{\text{comp}}$  and  $V_{\text{comp}}$  respectively).

### 3. Pilot analysis, constraints and degree of freedom

#### 3.1 Solid behaviour analysis

The solid mean residence time ( $\tau_s$ ) can be expressed by the sum of the time of circulation of the solid and the time spent in each compartment:

$$\tau_s = \frac{H_{\text{column}}}{v_{\text{imp}}} + N_{\text{imp}} \cdot T_m \quad (1)$$

where  $H_{\text{column}}$  is the height of the column and  $v_{\text{imp}}$  is the velocity of the particles during the impulsion.  $N_{\text{imp}}$  is the number of impulsions after which solid exits the column. In the case of a plug flow transport and if we assume that the solid is driven by the liquid impulsion, it can be expressed as a function of the column height and the height crossed during one impulsion:

$$N_{\text{imp}} = \frac{N_{\text{comp}} \cdot H_{\text{comp}}}{v_{\text{imp}} \cdot T_{\text{imp}}} \quad (2)$$

Then it comes:

$$\tau_s = \frac{N_{\text{comp}} \cdot H_{\text{comp}}}{v_{\text{imp}} \cdot T_{\text{imp}}} \cdot (T_{\text{imp}} + T_m) = \frac{N_{\text{comp}} \cdot H_{\text{comp}}}{v_{\text{imp}}} \cdot \left(1 + \frac{T_m}{T_{\text{imp}}}\right) \quad (3)$$

This last equation can be interpreted as:

$$\tau_s = \text{solid\_residence\_time\_without\_mixing\_step} \cdot \left(1 + \frac{T_m}{T_{\text{imp}}}\right) \quad (4)$$

During the mixing step, the solid suspension in the compartments is homogenised, generating intrinsic axial dispersion. Furthermore, this intrinsic axial dispersion will be increased if the volume of impulsion is different from the volume of one compartment. Finally, the axial dispersion will depend on the characteristics of the solid flow during the impulsion step.

#### 3.2. Constraints on the operating parameters

Consequently, in its conception, the pilot generates some more variables which act directly on the solid mean residence time independently of the global solid or liquid flowrates (i.e.  $T_{\text{imp}}$ ,  $T_m$ ,  $Q_{\text{imp}}$ ). But, of course, some constraints exist:

- A and f have to ensure a correct mixing of the particles in the compartments at the desired solid holdup;
- during the mixing step, the sphere must not be raised by the liquid;
- the volume of the impulsion must be as closed as possible to the compartment volume;
- the impulsion liquid flow must ensure a plug flow behaviour of the solid;

- the global liquid flowrate inside the column which has two contributions (the impulsion and the basic downward liquid flow rate) is constrained to respect the countercurrent flows functioning.

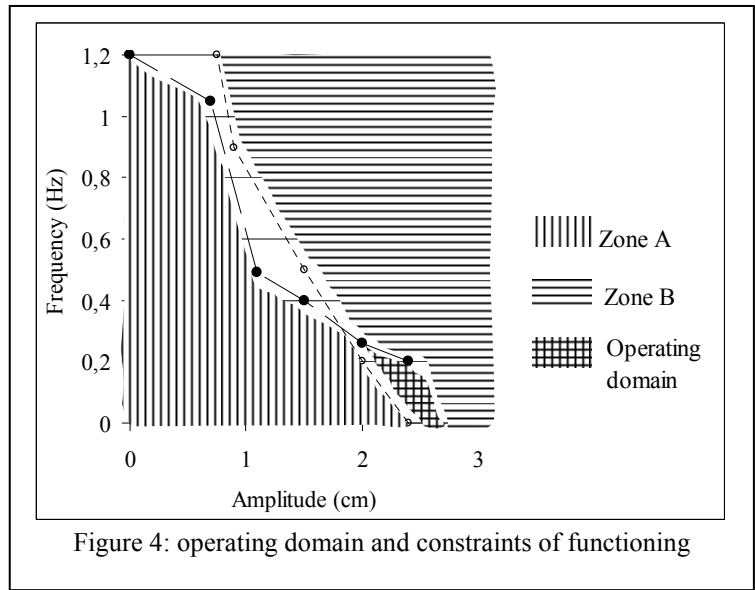
Considering that the inlet solid and liquid flowrates are fixed by the application, the different free variables mainly influencing the solid residence time are then  $T_m$  and  $T_{imp}$ . In comparison with traditional pulsed columns, this allows to control the solid residence time independently of the liquid.

#### 4. Pilot characterisation

##### 4.1. Operating domain for the mixing step

The first part of this work has consisted in finding a good experimental domain. For the considered solid (polystyrene powder with a  $1050 \text{ kg.m}^{-3}$  density and  $200 \text{ }\mu\text{m}$  in diameter), the characteristics of the pulsation, the frequency  $f$  and the amplitude  $A$  have to correctly mix the solid in each compartment without raising the spheres.

In a frequency/amplitude graph, the domains where the conditions are true have been established (Figure 4). Zone A corresponds to the correct sealing of the compartments while zone B to the good mixing of the two phases. The final operating domain is then the intersection of the two zones.



It is possible to predict this operating domain by the use of the terminal velocity of the particles  $u_t$  which is defined by:

$$u_t = \sqrt{\frac{4}{3} \cdot \phi \cdot g \cdot \frac{\Delta \rho}{\rho_L} \cdot \frac{1}{C_w}} \quad (5)$$

where  $\phi$  is the diameter of the particles;

$\Delta \rho$  the difference of density between solid and liquid phase;

$\rho_L$  the liquid phase density;

and  $C_w$ , the drag force coefficient.

Kunii et Levenspiel [7] proposed some relations to evaluate the drag force coefficient such as:

$$C_w = \frac{10}{\sqrt{\text{Re}_t}} \quad (6)$$

This relation is used under the condition:  $0.4 < Re_t < 500$ .

Then, the final expression of the terminal velocity of the particles is:

$$u_t = \left[ \frac{4 \cdot (\Delta \rho \cdot g)^2}{225 \cdot \rho_L \cdot \mu_L} \right]^{1/3} \cdot \varphi \quad (7)$$

with  $\mu_L$ , the viscosity of the liquid phase.

This calculation has been applied for three particles population: 900 $\mu$ m and 200 $\mu$ m polystyrene powder (PS) with a 1050 kg.m<sup>-3</sup> density and 135 $\mu$ m polychlorure vinyle (PVC) particles with a 1400 kg.m<sup>-3</sup> density. Figure 5 shows the calculated and experimental points.

This figure shows that the amplitude and frequency needed to perform a good mixing are more important for system with a high density difference between solid and liquid phases or for larger diameter. The experimental points confirm this observation but are not in perfect accordance to the calculation. Of course, this is due to the hypothesis that the mixing criteria is  $f \cdot A = u_t$ . Indeed, in a pulsed system, the hydrodynamic is very complex and is always in transient regime. This implies that the dynamic aspect (the acceleration) has to be considered. Nevertheless, this simple approach can be considered as a good tool to evaluate the possible operating domain of functioning of such a system.

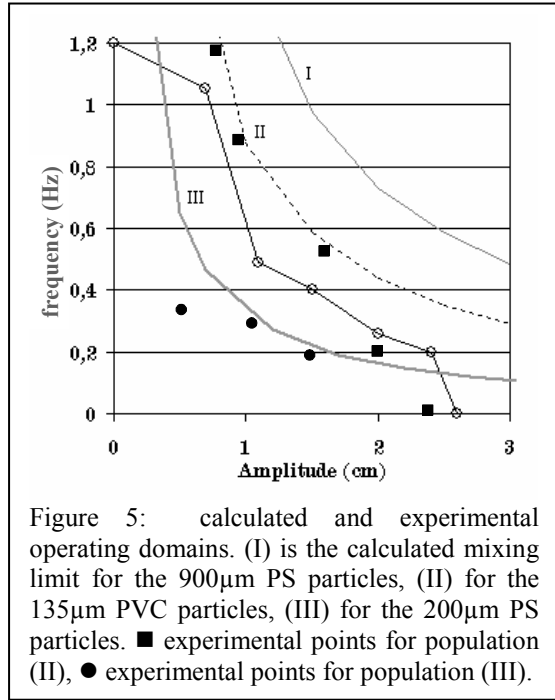


Figure 5: calculated and experimental operating domains. (I) is the calculated mixing limit for the 900 $\mu$ m PS particles, (II) for the 135 $\mu$ m PVC particles, (III) for the 200 $\mu$ m PS particles. ■ experimental points for population (II), ● experimental points for population (III).

#### 4.2. Solid phase residence time distribution

The objective of this study on the solid phase is to determine the influence of the pulsation and impulsion parameters on the transport of the solid. With the device described before, experiments have consisted in introducing a mass of solid in the lowest compartment of the column. The cake thickness is approximately equal to 1 cm in each trial set. Pulsation sets in suspension the particles. The slurry is then recovered continuously at the top of the column. Series of samples are finally filtered, dried and weighed.

In the present study, the solid tested was polystyrene powder ( $\rho=1050$  kg/m<sup>3</sup>) distributed in two classes of particles: 200 and 900  $\mu$ m. At the end of each test, the dried solid is sieved to separate fines and coarse particles.

Separating particles of different sizes could provide the distinct residence time distributions (RTD) of fines and coarse particles. In order to compare experimental data, each mass of solid is normalized by the total mass of the solid recovered at the outlet. Then, the solid mean residence time ( $\tau_s$ ) and the number of Peclet (Pe) are calculated by the classical momentum method. Because the total time spent by the solid inside the column can be chosen freely in function of  $T_m$  and  $T_{imp}$ , the solid RTD is presented in function of the number of impulsions or in function of the cumulated flowing volume.

Various duration and flow rate impulsions were tested. The operating conditions are:

Amplitude A: 1.7 cm

Frequency f: 0.3 Hz

$Q_{imp}$ : 200 - 300 L/h

$T_{imp}$ : 1 - 3 s (i.e. the volume fraction crossing the compartment varies from 0.3 to 1)

Figure 3 represents the influence of the volume crossing the compartments on the RTD. It shows that the axial dispersion increases when the impulsed volume decreases.

In the ideal case of an impulsion with a plug flow behaviour, if the volume going from one compartment to another is not equal to the volume of the compartment, this will induce an apparent axial dispersion. Indeed, when the compartments are sealed again (during the mixing step) the solid is “diluted” in the total volume of the compartment. In the case of a non plug flow behaviour, this intrinsic axial dispersion is added to the dispersion of the impulsion flow.

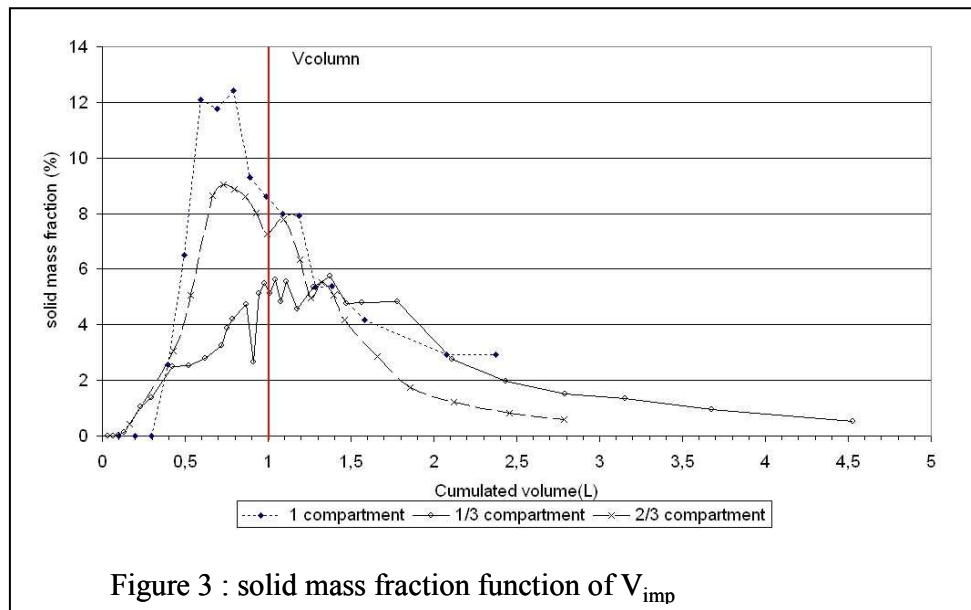


Figure 4 represents the influence on the RTD of the liquid flow rate during the impulsion (with a impulsion volume equal to one compartment volume). It shows that this flow rate has no significant influence on the solid behaviour. In fact, this result certainly depends on the particles stokes number in the liquid. Due to the pulsation, the solid tends to follow the liquid flow in the presented experiments.

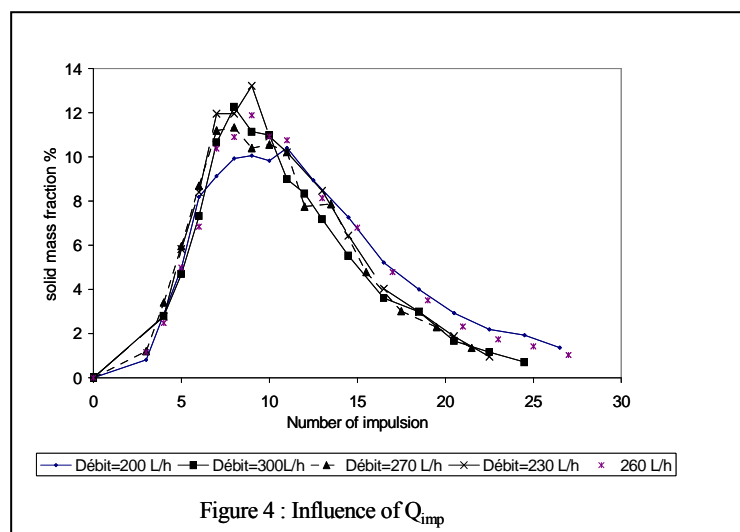
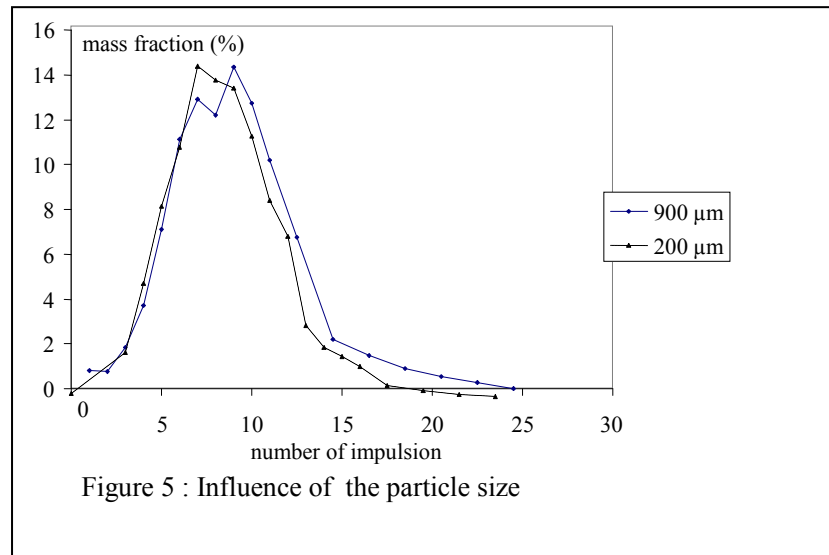




Figure 5 represents the influence of the size of the particles on the solid RTD. No significant difference can be observed, even if the larger particles seem to stay slightly longer in the column (for obvious reason). The main reason is that during the impulsion step, the liquid flowrate is very important and generates a high speed velocity in the column. Then the particles, what ever are their characteristics, followed the liquid phase.



#### 4.3. Conclusion on the pilot

The experimental study has shown that the pilot presents the following characteristics:

- a functioning with two separated and controlled steps (one for mixing and the other to transport the solid) is technologically possible;
- solid phase behaviour is not far away from a plug flow, with a mean residence corresponding to the number of physical stages in the column;
- fine and coarse particles have the same hydrodynamic behaviour and then no size segregation is observed;
- the objective of adding a degree of freedom is achieved: in order to increase the solid mean residence time without any changes on the liquid mean residence time, it is now possible to act on the mixing time ( $T_m$ ).

### 5. Solid-liquid application

The objective of this chapter is to determine the operating fields of the described apparatus and determine which unit operation can be performed in it. In order to limit our study, it has been decided to treat the case of a fixed column (in height and diameter). The comparison will be done with a classical pulsed column on the base of the mean residence time.

#### 5.1. Estimation of the solid residence time

In a first approach, the solid residence time in the pulsed column depends on three main parameters:

- the terminal velocity of the particles, linked to the drag force coefficient (through correlations of Kunii and Levenspiel 1969 for instance). The real velocity of the particles in the column ( $v_s^*$ ) has to be deduced from the previous velocity, modified

- by a coefficient which takes into account the geometry of the internals, the solid holdup and the frequency and amplitude of the pulsation (Haunold 1991);
- the velocity of the liquid phase, crossing the column in counter current;
- the height of the column.

So, the mean residence time will be somehow of the form:

$$\tau_s = \frac{H}{(v_s^* - v_L)} \quad (5)$$

Where  $v_s^*$  is the solid velocity without the liquid phase and  $v_L$ , the counter current liquid velocity.

The solid residence time in the BPC column can be expressed by the equation (3).

For both columns, the solid holdup is linked to the solid residence time and the volume of the column with the equation.

$$\Phi = \frac{Q_s \cdot \tau_s}{V_{column}} \quad (6)$$

where  $Q_s$  is the solid flowrate in volume.

## 5.2. Constraints on functioning

Of course, in the classical pulsed column, to avoid flooding,  $v_L$  must be lower than  $v_s^*$ . If the treated solid is polydispersed, all particles for which  $v_s^*$  is smaller than  $v_L$  will be driven towards the top of the column. However, if a high solid mean residence time is needed, the operator has to increase the liquid flowrate and to operate close to the flooding point. In this case, it will increase the segregation and the system will be very difficult to control.

In the BPC column, the main constraint is on the countercurrent condition: the global liquid flowrate circulating through the column must be downward. This global flowrate has two contributions:

- the liquid introduced continuously at the top of the column;
- the liquid introduced at the bottom of the column during the impulsion step.

This condition can be written:

$$Q_L \cdot \left( 1 + \frac{T_m}{T_{imp}} \right) > Q_{imp} \quad (7)$$

where  $Q_L$  is the liquid flowrate in volume introduced at the top of the column.

Equations (3) and (7) imply that for very short solid residence times,  $T_m$  must be low and the inlet liquid flowrate must be high. But as equation (7) is an inequality, it means that the possible operating domain is important.

## 5.3. Example of calculation

The solid residence time, the holdup and the liquid flowrate have been calculated for a column with the following characteristics:

- 0.1 m in diameter;
- 1 m of height;
- 0.1 m of height for each compartment (from disc to disc in the classical pulsed column);
- an amplitude of 0.02 m and a frequency of 2 Hz has been chosen for the pulsation in order to ensure a good mixing of the particles;
- an impulsion liquid flow rate of 800 L.h<sup>-1</sup> has been chosen for the BPC column.
- particles of 700µm in diameter and 1.4 kg.L<sup>-1</sup>.

Figure 6 presents the obtained results. It shows the operating domain (in grey) and the constraints in function of the desired solid residence time. Over point A, the BPC column allows the choice of the liquid flowrate over (zone I) or under (zone II) the liquid flowrate of the classical column. Of course, this choice will be made as a function of the process objectives. For solid residence time under point A, the range of liquid flowrates for the BPC column is much more restrictive.

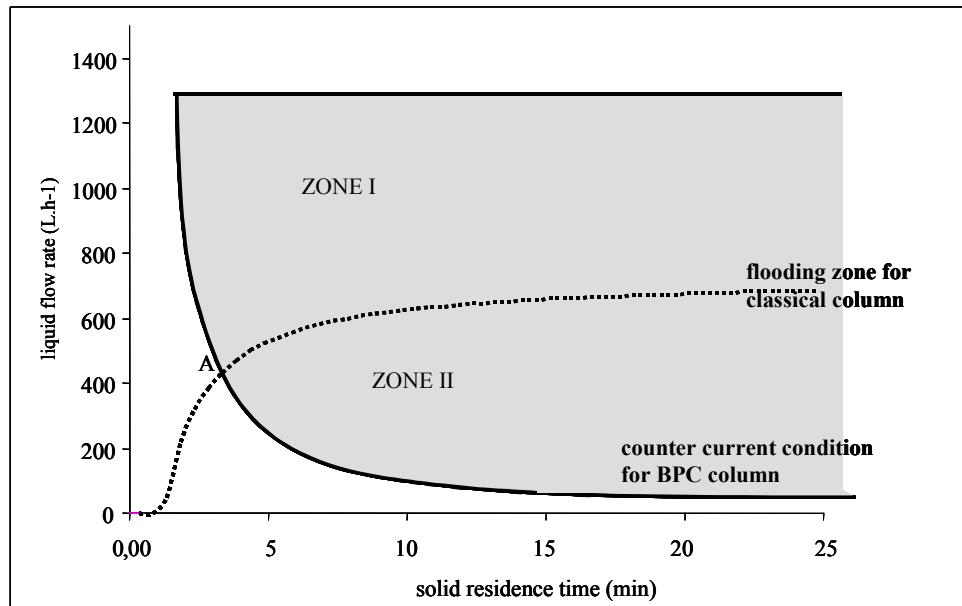


Figure 6 : Operating domain

Despite of the important hypothesis and simplification of the previous calculation, this figure allows to clearly define the possibilities and limitations of this new solid-liquid column.

## 6. Conclusion

In this study, a new kind of solid-liquid contactor has been designed, built and tested, the BPC column. The main objective is to enhance the possibility of such a contactor via modification of the internals and via non sinusoidal pulsation. Indeed, in classical pulsed column, the solid mean residence time is coupled with the liquid flow behaviour.

The experimental study has shown that it is possible to operate with two separated and controlled steps (one for mixing and the other to transport the solid). The use of physically separated compartment allowed reducing the constraint between the two phase flows. Furthermore, the difference in behaviour between large and small particles seems to be much less important with this new apparatus. Finally, even if this study also shows that this new column presents less flexibility when low residence time is needed, this column should be useful for a lot of applications for which solid residence time must be long (for example in solid-liquid extraction). This work can then be considered as an example of process intensification, by the use of a new technology able to decrease either the solvent consumption or the volume of the column.

## Symbols used

$A$	amplitude of the pulsation	[cm]
$f$	frequency of the pulsation	[Hz]
$T_m$	mixing duration	[s]
$T_{imp}$	impulsion duration	[s]
$Q_{imp}$	impulsion flowrate in volume	[L.h <sup>-1</sup> ]
$Q_L$	liquid flowrate in volume at the top of the column	[L.h <sup>-1</sup> ]
$Q_S$	solid flowrate in volume	[L.h <sup>-1</sup> ]
$\Phi$	solid fraction holdup	
$V_{column}$	total column volume	[L]
$\tau_S$	mean solid residence time	[s]
$u_t$	terminal velocity of the particles	[cm.s <sup>-1</sup> ]
$v_{imp}$	velocity of the particles during the impulsion	[cm.s <sup>-1</sup> ]
$v_S$	solid velocity	[cm.s <sup>-1</sup> ]
$v_L$	counter current liquid velocity	[cm.s <sup>-1</sup> ]
$\mu_L$	the viscosity of the liquid phase	[Pa.s <sup>-1</sup> ]
$\rho_L$	liquid phase density	[kg.m <sup>-3</sup> ]
$\phi$	diameter of the particles	[m]
$\Delta\rho$	difference of density between solid and liquid phase	[kg.m <sup>-3</sup> ]
$C_W$	the drag force coefficient	
$N_{comp}$	number of compartments	
$H_{comp}$	compartment height	[cm]
$V_{comp}$	compartment volume	[L]
$H_{column}$	column height	[m]
$N_{imp}$	number of impulsions before the solid exits	

## Acknowledgement

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