Use of the pulsation to control a polydispersed particles flow in a new type of pulsed column

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Abstract:

New internals have been designed and implemented in a semi-industrial continuous solid-liquid pulsed column. By coupling these internals with a non sinusoidal pulsation, a better control of polydispersed solid particles is achieved. The pulsation is composed of a mixing step during which only the liquid flows through the column and an impulsion step designed to transport the solid phase from stage to stage.
Solid and liquid phase behaviours are characterized thanks to residence time distribution measurements. This study demonstrates the strong impact of the pulsation and of the liquid flow rate during the impulsion to reduce the particles segregation inside the pilot. The effects of operating parameters on the liquid phase are also investigated and the choice of an operating compromise is discussed to balance advantages and drawbacks on the process.

1 Introduction

Pulsed columns are an interesting tool among a wide variety of solid liquid contactors. Its main advantages lean on the continuous functioning, the counter-current mode and the suspension of particles. However, the operation of these columns remains difficult as they often meet flooding problems, fines driving and too short residence times. In a general way, solid circulation is not well controlled, or is narrowly linked to the liquid flow [1, 2, 3].
When the kinetics of mass transfer or reaction occurring in the column is rather long (above a few minutes), a too short residence time of solid will either be compensated by the conception of a high column, or either by enhancing the liquid flow, which will increase the consumption of solvent and the quantity solid losses because of fines driving.

Up to now, pulsation was only used to slow down the drop of particles and to ensure an intimate contact between solid and liquid phases in continuous pulsed columns. In this work, new internals and a new pulsation mode lead to a fine tuning of working conditions: better control and reduction of dispersion of polydispersed particles flow.

In previous works, we developed a system in which the solid can be stopped in compartments while the liquid continues to flow [4]. This has been obtained by the use of simple rustic mechanic valves on a porous media through which the liquid can flow. In the other hand we used a non sinusoidal pulsation developed and studied for solid-liquid systems [5]. This pulsation is used to enhance the mixing of the two phases and to open the compartments and transport the solid phase.

In this study, we enhanced the used technology and made it more reliable and flexible [6]. Indeed, on the previous apparatus, the opening of the valves was directly linked to the liquid flow rate which was used to rise up the bullets and then to open the compartments for the solid flowing. The use of pneumatic valves allows completely decoupling the flowing of the solid from the liquid pulsation.

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, results concerning the solid and liquid residence time are presented. The global transport of the two phases will be analysed, so will be the impact of this technology on the solid granulometric segregation.

2 Material and methods

2.1 Pilot description

Solids and mother liquid enter continuously at the bottom of the column; whereas the purified suspension overflows at the top through an outlet tube (see Figure 1). The washing solvent is injected at the top of the column and flows downwardly.

The active zone of the column is composed of stacked compartments, which are separated by a filtering cloth and a pneumatic valve. The configuration of a compartment is detailed in figure 2. The filtering cloth allows the solvent flow and retains solid particles. The solid suspension is only enabled to circulate through the valve pneumatically controlled. A disc is placed above the valve in order to divide the jet of fluid coming from the orifice of the valve, positioned at the centre of the tray. Such a configuration enhances the formation of vortices between the baffles and the trays, around the valve.

The column operates on a cycle mode with two sequences:
(i) a mixing step; a classic sinusoidal pulsation is used to mix the two phases in each compartment; the valves are closed and the particles are trapped between two trays while the liquid phase is flowing downwardly on average; this step is characterized by the amplitude A and the frequency f of the pulsation which has to be intensive enough to generate high speed vortices, able to drive dense particles. This step is programmed with a timer, during a fixed duration T_{mixing}.
(ii) an impulsion step; this second step allows for a brief upward flow of high velocity while the valves are opened and the pulsation is still generated. This impulsion is dedicated to the solid transport from one compartment to the next. It is characterized by its flow rate Q_{imp}, the duration T_{imp}. The volume injected is written V_{imp}.

2.1 Experiments

The objective of this study on the solid phase is to determine the pulsation and impulsion parameters leading to a plug-flow 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 2 cm in each trial set. Pulsation sets in suspension the particles. Here, the column does not operate on a cycle mode: an upward flow produced continuously has been preferred to characterise the transport of the solid during the impulsion step. The slurry is then recovered continuously at the top of the column while the valves are permanently opened. Series of samples are finally filtered, dried and weighed.
In the present study, the solid tested was polystyrene powder (d=1050 $kg.m^{-3}$) distributed in two classes of particles: 200 and 900 µm. At the end of each test, the dried solid is sieved to separate fines and coarse particles.

Various operating conditions have been tested. The variation ranges are:

Amplitude A : 0.6 - 3 cm

Frequency f : 0.39 - 1.35 hz

Q_{imp} : 400 - 700 $L.h^{-1}$

Separating particles of different sizes could provide distinct residence time distributions (RTD) for 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 (Ts) and the number of Peclet (Pe) are calculated by classical momentum method.

The liquid phase residence time distribution experiments were performed by injecting 15 mL of sodium hydroxide in an injection loop added to the liquid inlet. Two three-way valves are turned simultaneously in order to let the liquid feed sweep the volume occupied by the tracer. Injections take approximately 15 s, to be as close as possible to a perfect pulse injection in comparison to the time scale of the experiment (1500-2500 s). The conductivity probe versus time is then measured directly at the exit by means of a conductivity probe.

The output concentration deduced from the conductivity is normalized to obtain the exit age distribution.

3 Results and discussion

3.1 Solid behaviour

Figure 3 presents the fraction of outlet solid function of the normalized time for the two particles classes and for various operating conditions.

The absence of oscillations or small oscillations were found to segregate the solid flow of a bimodal population. For example, a pulsation with A=2.4 cm and f=1Hz superimposed on a unidirectional flow at 400$L.h^{-1}$ was sufficient to allow a synchronous outlet of particles of all sizes. These oscillatory conditions correspond to an oscillatory flow velocity of 2.4 $cm.s^{-1}$, compared to the terminal velocity of 1.4 $cm.s^{-1}$ for coarse particles and of 0.3 $cm.s^{-1}$ for fine particles.

On the contrary, at 0.75 Hz and 1.8 cm, the measurement of mass fractions showed that fines particles were transported faster than coarse ones. And it was observed visually that a part of solid, mostly 900 µm, stayed towards the base end of the column. At high oscillatory velocities, the combined effect of the baffles and the pulsation causes the apparition of intense recirculations occupying the whole volume of the compartment, and reducing dead volumes. When the velocity in such vortices is equivalent to several times the terminal velocity, the particles are driven in recirculations, and maintained in suspension. The solid phase forms nearly uniform slurry that will be transported upwardly thanks to the impulsion. The evolution of solid mean residence time (figure 4) confirms that when the pulsation increases, the differences between 200µm and 900µm particles Ts is reduced. These results are in good agreement with the study performed by Mackley et al. who determined that pulsation was a powerful tool to control segregation in closed pulsed columns [2].

The same kind of behaviour was observed concerning the influence of the impulsion flow rate. In such conditions, the system operates closer to a plug flow for greater upward flowrate. Experiments carried out with bimodal populations also demonstrated that granulometric dispersion was reduced for high flowrate. Enhancing the unidirectionnal flow destinated to the transport of the solid tends to increase the size and the intensity of recirculations.

Finally, experiments have been performed to characterize the impact of the impulsion volume (Af = 1.8 $cm.s^{-1}$, Q_{imp} = 500 $L.h^{-1}$, V_{imp}=0.4 or 0.9 or 1.5 times the compartment volume). From Figure 5, presenting the residence time distribution, it is possible to confirm that in suitable conditions of agitation of the particles, the impulsion volume has very little influence on the particles behaviour.

3.2 Liquid behaviour

The specific effects of frequency and amplitude of pulsation on the liquid behaviour was also examined. A partial set of RTDs performed for different frequencies are presented in figure 6. From figure 7, a net decreasing tendency of Peclet number in function of the intensity pulsation can be observed. Indeed, pulsations enhance axial mixing in the liquid phase, by increasing the size and the intensity of the vortices, all the more so as the radial convection is limited by tube walls. The opposite results were shown in previous paragraph concerning the solid phase. Based on our observations and experiments on bimodal populations, the physical explanation is provided by the fact that particles need a minimum velocity to be transported. Solid suspension needs intense recirculations occupying the whole compartment to be driven. Furthermore and unlike the liquid phase, solid phase back-mixing is limited because particles are trapped between two trays.

It is obvious that the introduction of the impulsion step will have a negative impact on the liquid flow. The upward flow is opposite to the average counter-current and downward liquid flow. Back-mixing is inherent to our system. This phenomenon can be observed on figure 8. The tracer concentration was followed at the liquid outlet for different mixing time. One experiment was also performed with no impulsion at all (T_{mixing}=infinite). It is interesting to notice the peaks due to the repeated impulsions. And, as expected, the dispersion of residence time distribution is increased when impulsions occurs more frequently.

An equivalent series of experiments has also been performed to examine the effect of the volume impulsed. The results are not shown here, but, in the same way, it demonstrated that the increase of volume impulsed enhanced back-mixing.
Lastly, the investigation on the impulsion flowrate found no influence on the liquid behaviour.

3.3 Global behaviour

Not enough experiments have been performed to define a precise criterion which will characterize the impulsion flow rate and the intensity pulsation necessary to allow a plug flow transport of the solid. It is also too early to uncouple the role of both parameters. However, it results from these experiments that it is essential to generate an intensity pulsation and impulsion flow rate superior or equal to the terminal velocity of the heaviest particles.
In the operation mode of the column, pulsation is the base for mixing the particles in the compartment and enhances mass transfer between the suspension and the solvent, during the mixing step. It is now established that pulsation is also a precious tool to improve RTD performance and to allow more flexibility in the practical choice of the impulsion flow rate.
A series of experiments designed to test the role of the volume injected on the solid flow found no influence for a range between one third and one compartment and a half, provided impulsion and the pulsation are powerful enough.

In this column, the main constraint lies on the counter current condition: the global liquid flow rate circulating through the column must be downward. This global flow rate has two contributions: (i) the liquid introduced continuously at the top of the column; (ii) the liquid introduced at the bottom of the column during the impulsion step. The upward flow is opposite to the average counter-current and downward liquid flow. Liquid back-mixing is then inherent to our system. This drawback has then to be taken into account when designing the column, choosing the operating parameters and evaluating the process.

4 Conclusion

A new technology has been set up in order to improve continuous columns used in solid-liquid contacting. An important aspect of the innovation is the introduction of a step mixing which allows almost controlling the solid residence time. Another aspect is the role of pulsation for an homogeneous transport of the solid suspension, in particular, the reduction of the granulometric dispersion.

Residence time distribution (RTD) experiments for both liquid and solid phases have been performed. The study allows concluding:
(i) a good pulsation is necessary to minimize the axial dispersion for the solid phase. The needed intensity increases when the impulsion flow rate is lower. When the solid phase is not correctly agitated, the fine and coarse particles have different mean residence times in the column. This effect is inverse then the one observed on liquid phase in classical pulsed column, but confirms the observation of Ni et al [7, 8] on the solid segregation in disk and doughnuts pulsed columns.
(ii) impulsion flow rate has the same impact on the solid behaviour as it increases the velocity of the internal recirculation loop.
(iii) no real influence of the impulsion volume has been observed when the solid is correctly agitated.
(iv) effect on the liquid phase of the operating parameters remains classical. The main drawback is that back mixing increases with the pulsation intensity.

Based on the experiments presented, this column would be valid for dense particles presenting a low difference density with the liquid phase. The column has also been tested for floating particles and applications can possibly be extended to more dense solids, provided a powerful system of pulsation can ensure high velocities in the column.
For the column used in this study, mixing time ranges between 2 and 3 minutes and impulsion time between 5 and 10 seconds, which corresponds to residence times around 20 minutes. But, the counter-current condition makes the use of the new technology suit the type of system where a long residence time is needed.
A wide scope of potential applications is apparent: heterogeneous reactions, washing limited by diffusion. Of course, for high concentrations and dense particles, added complications of having to maintain in suspension the particles will be considered.

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Symbols used

A	amplitude (cm)
f	frequency (Hz)
T_{mixing}	duration of the mixing step (s)
Q_{imp}	liquid flow rate of the impulsion step ($L.h^{-1}$)
T_{imp}	duration of the impulsion step (s)
V_{imp}	injected volume during the impulsion step (L)
Ts	the solid mean residence time (s)
Pe	Peclet number

Figure 1: scheme of the device and process

Figure 2: pilot internal scheme and picture. (1) pneumatic valve, (2) passage for the solid phase, (3) valve actuator, (4) filtering cloth to retain the solid phase, (5) disc.

Figure 3: particles RTD (age versus normalized time) for 200µm diameter particles (○) and 900µm diameter particles (◆). Q_{imp} = 400 $L.h^{-1}$ and Af=1.8 $cm.s^{-1}$ for figure (a) and Af=0 $cm.s^{-1}$ for figure (b). Q_{imp} = 700 $L.h^{-1}$ and Af=1.8 $cm.s^{-1}$ for figure (c) and Af=0 $cm.s^{-1}$ for figure (d).

Figure 4: Solid particles mean residence times: function of Af (● for Q_{imp}=400$L.h^{-1}$ and for 900µm particles; ○ for Q_{imp}=400$L.h^{-1}$ and for 200µm particles; ▲ for Q_{imp}=700$L.h^{-1}$ and for 900µm particles; △ for Q_{imp}=700$L.h^{-1}$ and for 200µm particles)

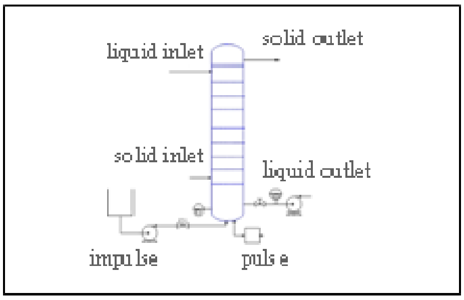
Figure 5: RTD function of normalized time at Af=1.8 $cm.s^{-1}$, Q_{imp} = 500 $L.h^{-1}$. (△ for V_{imp} = 0.4 V_{comp} ; ◆ V_{imp} = 0.9 V_{comp} and x for V_{imp} = 1.5 V_{comp})

Figure 6: Liquid RTD function of Af ; Q_{imp}=700 L/h ; V_{imp}=830mL ; T_{mixing}=160s. (a) Af = 1.8 $cm.s^{-1}$; (b) Af = 0.75 $cm.s^{-1}$; (c) Af = 0.6 $cm.s^{-1}$; (d) Af = 0.25 $cm.s^{-1}$.

Figure 7: The dependency of the Peclet number on the pulsation intensity Af (cm/s)

Figure 8 : Measurement of the normalized concentration in tracer at the liquid outlet ; Qimp=600L/h ; Vimp=840 mL ; Af=2.3 cm/s ; Influence of the duration of the mixing step

Figure 1



liquid inlet
solid outlet
solid inlet
liquid outlet
impulse
pulse

Figure 2

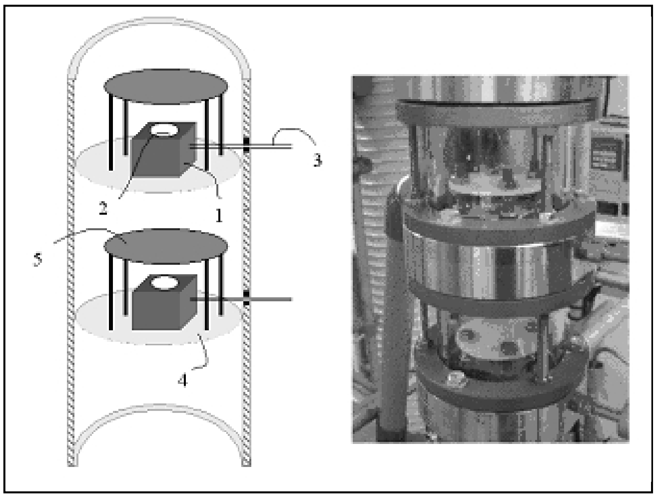


Figure 3

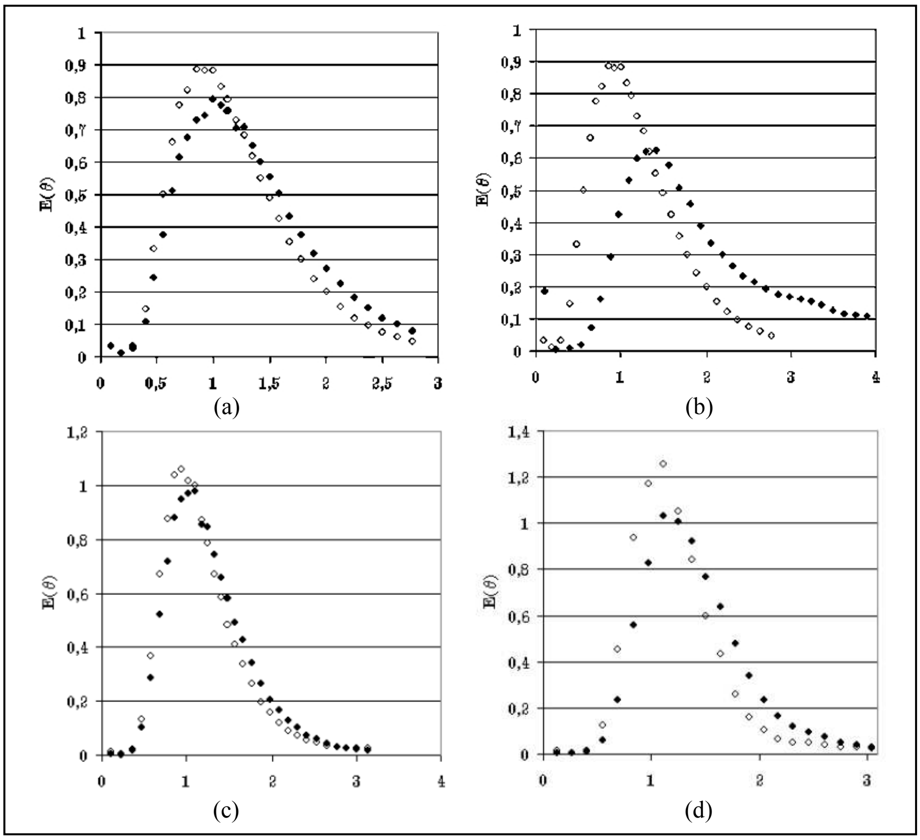


Figure 4:

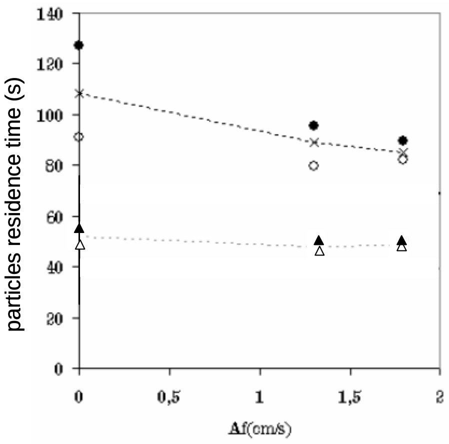

particles residence time (s)
140
120
100
80
60
40
20
0
0
0,5
1
1,5
2
Af(cm/s)

Figure 5

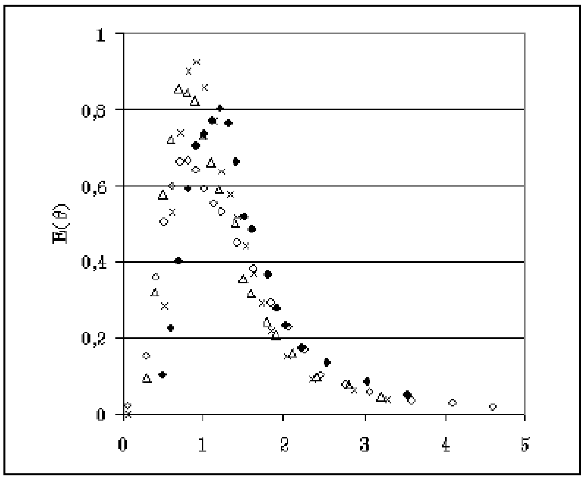
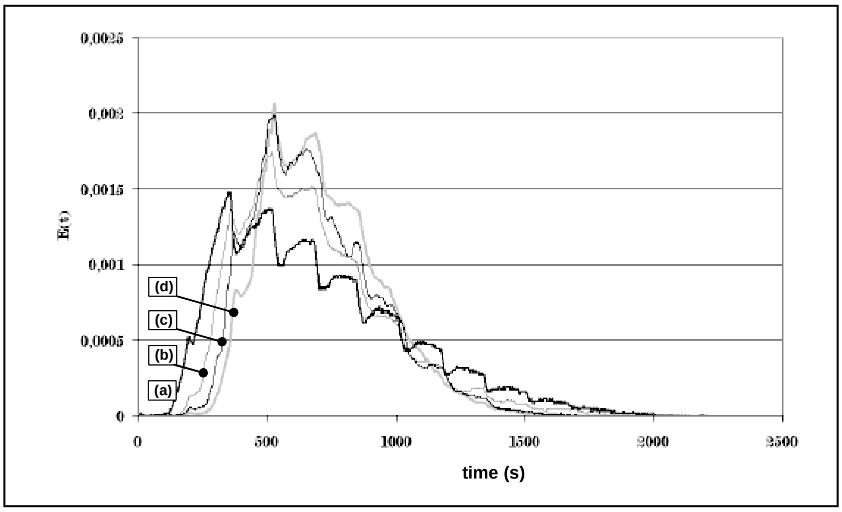


Figure 6



0,0025
0,002
0,0015
0,001
0,0005
0
E(t)
(d)
(c)
(b)
(a)
0
500
1000
1500
2000
2500
time (s)

Figure 7

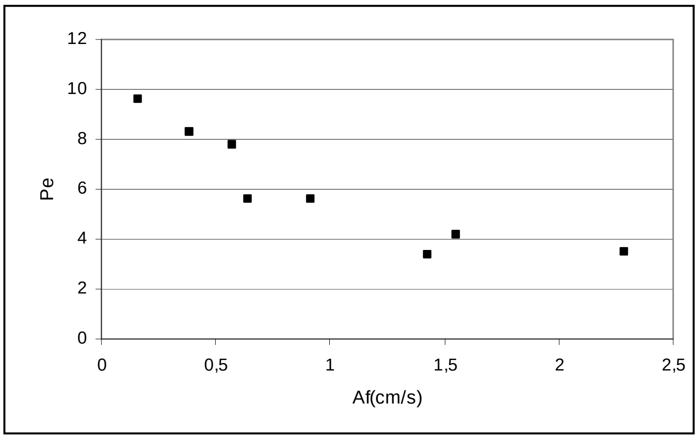
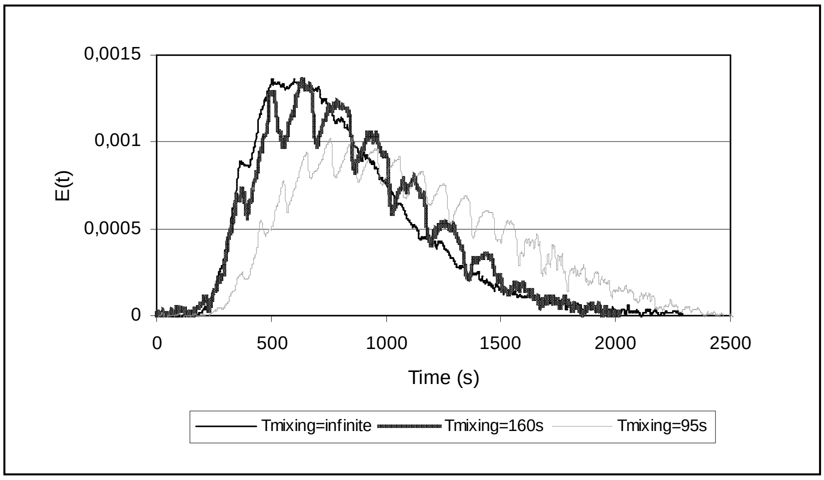


Figure 8



0,0015
0,001
0,0005
0
E(t)
0
500
1000
1500
2000
2500
Time (s)
Tmixing=infinite
Tmixing=160s
Tmixing=95s