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A New Pulsation Policy in Disc and Doughnut Pulsed Column Applied to The Solid-Liquid Extraction of Andrographolide from Plants.

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Abstract:

Classically, sinusoidal oscillations are imposed to enhance mixing and mass transfer between two phases contacted. 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. The objective of this study is to demonstrate the viability of using a non-sinusoidal pulsation in a continuous contactor to replace a traditional batch sinusoidal mode. A review of the different pulsation techniques is firstly presented. The example of solid-liquid extraction of Andrographolide from plants has then been chosen to bring out the advantages of the new pulsation mode. The development of this application as a continuous process in a column has indeed encountered difficulties due to the important heterogeneity of the matter : one of these classes tends to float and the other to sink, which always leads to a definitive flooding in classical operations. Typically, the proposed signal is composed of two different periods : in one hand, a classical sinusoidal pulsation step used to mix the liquid and solid phases in the active part of the column and allow an optimal mass transfer and, in a second hand, an impulsion phase, used generally for the transport of solid. The extraction is carried out in a disc and doughnut column of 54 mm in diameter, and 3.5 m in height. Liquid and solid are flowing cocurrently and downwardly. Experiments have been performed to know the global characteristics of the process in steady state and to suggest some elements for industrial design. The results showed that an optimal tuning of the geometric characteristics of the column, the level of interface and the parameters of the pulsation, could increase the operated domain where flooding is avoided.

Keywords: solid-liquid extraction, separation, pulsed column, non sinusoidal pulsation, andrographolide.

1 Introduction

Pulsations, in continuous processes, usually provide a good fluid mixing which ensures efficient mass and energy transfer. Pulsed flow offers an interesting alternative means of external agitation in columns used as liquid-liquid or solid-liquid contactors.

But, 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

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flooding problems, granulometric dispersion or short residence times can be met.

After a review of the pulsation techniques and policies, this article describes the new pulsation mode and illustrates its advantages through the example of the solid-liquid extraction of andrographolide from plants in a discs and doughnuts pulsed column.

2 Historical Development

Pulsing techniques

Pulsed column was first proposed in 1935 by Van Dijck and was dedicated to liquid-liquid extraction [1]. Oscillations were transmitted directly from a piston or bellows to the continuous phase in the column which contained stationary packing or plates (Figure 1). The first major application was in the nuclear industry for refining uranium by solvent extraction. The techniques of pulsation are primarily of two types, either an hydraulic system, or a pneumatic system with a pulse leg.

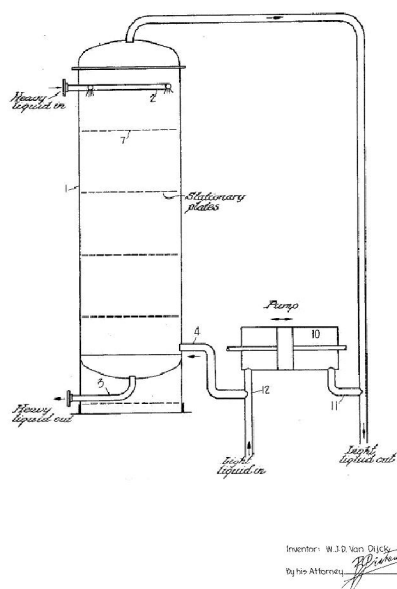


Figure 1 : First pulsing hydraulic system proposed by Van Dijck

The simplest hydraulic system is composed of a piston which communicates an oscillatory movement directly to the liquid in the column. The problem is that, in these systems, the

gasket which insulates the shaft actuating the piston is subjected to a strong wear. The process fluid can leak later. Moreover, the pressure necessary to generate the pulsation is important since it corresponds to the pressure to move the volume of liquid contained in the column. This problem is however solved if the piston is in vertical position in the pulse leg. Some of the processes in industry used stainless steel or PTFE bellows. A diaphragm pump can avoid the leak of process fluid in the hydraulic system. The configuration of this diaphragm pump, in which the valves can be removed, offers a satisfactory system for laboratory columns. Generally, hydraulic systems offer the advantage of reliability and ease of maintenance. The functioning cost and the limitation to small throughput of columns are its main drawbacks. However, it is a very suitable system for tests laboratory [2].

There are mainly two types of pneumatic systems: (1) those which use an incompressible fluid which controls the displacement of a piston; and (2) those which use a compressible fluid like air, inert gas, and which transmit the pulsation to the fluid by direct contact (air pulsing technique). This latter device is the most employed in nuclear industry as it avoids moving parts in or near the column. This is an important advantage because the contact maintenance would require decontamination from radioactivity and consequent interruption of plant operation.

The schematic of a typical air pulser system is illustrated in figure 2. The leg and the column constitute a U-tube with two dissymmetrical branches. The air pulser consists of a valve which admits air from the reservoir at constant pressure and a valve which exhausts air from the pulse leg. The adjustment of frequency of opening the valves permits to control the pulse frequency and the tuning of the pressure in the air reservoir sets the pulse amplitude. In figure 2, the valves are controlled by solenoid valves. Otherwise, other commands like rotating cams, or rotary valves can be set up [3].

These pulsers are considerably less expensive than piston pulsers [3]. As regards power consumption, air pulsing technique is only interesting when functioning at natural frequency. This concept was developed by Baird and Ritcey in 1965. The liquid, after a

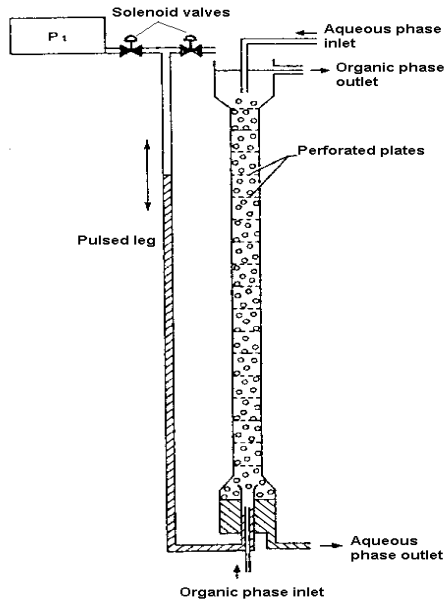


Figure 2 : Pneumatic pulsation system

first “drive”, is naturally subjected to oscillations, resulting from an energy balance between frictional losses of liquid against the leg wall, and the compressibility of the enclosed air space. At natural frequency, the air space can store and release enough energy in order to deliver smooth accelerations and decelerations of the liquid column. The amplitude can be tuned by acting on the pressure of the reservoir. The range is large and not limited when functioning at natural frequency. The range is more constrained for others frequencies, as it is explained at the end of this paragraph. In resonant conditions, the frequency is depending on the system (diameter of the pulse leg, nature of the liquid, nature of the gas). Otherwise, it is practically difficult to operate at non-resonant conditions : for frequencies weaker than natural frequency, the system will tend to oscillate at the natural frequency, whereas, past natural frequency, it has been noticed by Weech and Knight that the amplitude falls off for highest frequencies [4]. Indeed, as the exhaust valve is opened earlier, less volume of air is under pressure. A correlation calibrating the amplitude of the pulse, as a function of the frequency and the pressure of the reservoir is generally proposed. The correlation has the following shape [5]:

$$A = k_1 \cdot P^{k_2} \cdot f^{k_3}$$

where A is the amplitude of the pulsation, P the pressure of the air reservoir, f the frequency. k_1 , k_2 , and k_3 are constants experimentally determined.

In conclusion, functioning at natural frequency is very interesting on an energy and air saving point of view, but the frequency is fixed by the system. Out of these conditions, air-pulsing consumes more power than direct pulsing [3], and a compromise has to be found between amplitude and frequency, for highest frequencies.

Mode and role of pulsations

Hydraulic and air pulsers will classically give a sine wave shape to the column pulse, or triangular signals (constant speed), with a light damping. With specific cams for mechanical pulsers or with an adapted tuning of solenoid valves for pneumatic systems, square wave or dissymmetrical signals can be generated. Signals with less accelerations will always minimize pressure drop.

Typically, frequencies in the range 0.25-3 Hz and amplitudes in the range 0.25-3 cm were used, whatever the column diameter is. Fluid pulsation, in this range of frequencies has to be powerful enough to reach an emulsion-type operation of the column, that is to say that dispersed phase remains as droplets, in the case of liquid-liquid contact [6]. For solid-liquid contact, pulsation has to put and maintain in suspension the particles in the fluid phase. It has been largely demonstrated that increasing pulsation above this hydrodynamic optimum level reduced the contacting efficiency because of back-mixing increase [7].

Sometimes, more complex pulsation modes are implemented, particularly when the pulsation has a more extended role than setting in suspension the particles. For example, the CEA (Commissariat à l’Energie Atomique) patented a column with baskets and crowns in which the solid is subjected to a fast and weak pulsation intended for mixing, and then to a pulsation of very weak frequency, but of strong energy to force the solid phase towards the lower basket [8]. By this way, it is possible to regulate the rate/rhythm of drop of the solid phase, while maintaining conditions favorable to mass transfer. A second example has been noticed in the industry of fine chemistry : problems of filterability and inertia of beds of particles arise when washing crystals. Thus, a dissymmetrical mode of pulsation is recommended, with a strong acceleration of the signal to break the "cake" formed on the

filter, the movement going down being slower and less abrupt. Between each pulsation, solid elutriates during a rest period. Such a mode of pulsation tends to minimize the losses of energy. Finally, a study from Boyadzhiev and Angelino [9,10] leads to a model which could predict the speed of solid particles through a liquid flow, with different conditions of pulsation. It proved that, for a dissymmetrical signal with an acceleration stronger than deceleration, the dense solid could go up, by adapting amplitude and frequency. This means that it would be possible to slow down the solid while falling, or to re-examine the traditional direction of flow of the solid.

This short review of pulsations in pulsed columns shows the wide variety of techniques existing, and the different shapes of signals generated both in industry and in laboratories. It can be noticed that academic studies before 70's widely concerned the technological point of view, whereas more recent works consist in more theoretical analysis aiming to optimising amplitude-frequency combinations for a maximum efficiency. But, problems specific to solid phase, like flooding, clogging, or fines driving often lead to technological solutions either confidential or claimed in patents. Innovations in processes to improve solid transport are seldom subjected to a more theoretical study. Formalization of a new pulsation policy to develop a continuous process is proposed in the following sections.

3 Application of the pulsation policy to an example of solid-liquid extraction

In the studied case (extraction of active principles from plants), some problems raised from the heterogeneity of the vegetal material turning to the advantage of batch processes in comparison with continuous contactors. The difficulty lies in the completely different hydrodynamic behaviour of two classes of particles in the raw plant substrate. One of these classes tends to float and the other to sink. Function of the solvent concentration, the characteristic dynamics of behaviour changes but this always leads to a definitive flooding in classical extraction columns.

3.1 Plant description

Andrographis paniculata is an erect annual herb varying in height from 30 to 100 cm, 2-6

mm in diameter. It has been used as traditional medicinal plant in Southeast Asia, China and India. It makes a good treatment for infectious fever-causing diseases, clear heart, and antibacterial, diminish inflammation, removing toxins, arresting diarrhea, respiratory infections, and dysentery caused by bacterium. In Europe and in South America, people use the plant for common colds and sore throats [11]. The active components of *Andrographis paniculata* is andrographolide which is a very bitter compound known as diterpene lactones found in the aerial parts of the plant (leaves and stems). Andrographolide is an unsaturated trihydroxy lactone (C₂₀H₃₀O₅) [12,13].

The plants particles of this work can be separated into two shapes : cylinder refers to stems and plate refers to leaves. The diameter of leaves is 35 μm, and the diameter of stem is 900 μm, according to the analysis done with a Scanning Electronic Microscope. The density of this plant (ρ) and porosity (ε) are equal to 837 kg.m⁻³ and 0.33 respectively [14].

Analysis by high pressure liquid chromatography, after experiments in soxhlet reactor, determined that the concentration of andrographolide in leaves and stems are 44.38 mg/g of plant and 15.20 mg/g of plant respectively. The andrographolide concentration in leaves is then three times more important than in stems. Leaves in sample represent about 80% in mass of the total solid plant.

Experiments have been done to determine the evolution of the sinking mass fraction versus time, for different concentrations of ethanol in the solvent (95 %, 70%, 50% or 0% percentage of ethanol). Figure 3 shows that the sinking mass fraction increases with time, as porosity is filled up with the solvent. And the solvent absorption apparent kinetic increases with the percentage of ethanol.

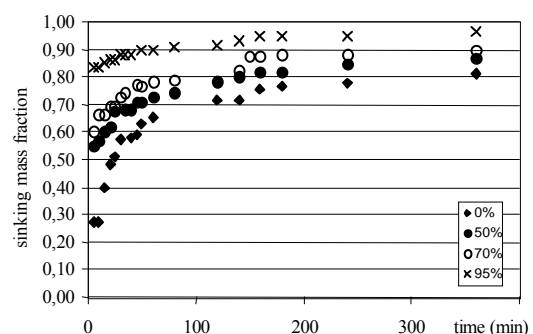


Figure 3 : Sinking mass fraction function of time for different concentration in ethanol

For the objective of a continuous process in a pulsed column, one main point is pointed out: it will not be possible to avoid a flooding of the column with a traditional functioning. Furthermore, it has been noticed that the floating solid is mainly composed of stems which do not contain the main part of the solute and that the percentage of ethanol has a significant effect on the sedimentation of the plant : the higher the percentage, the easier is the solid phase flow.

As a conclusion, it has been decided to propose a technological solution to separate, inside the pulsed column, the different parts of the solid phase. The objective is to eliminate at the top of the column the floating part. At the head of the column, a tube has been added in order to allow the purge of the floating part and a pneumatic pulsation system has been used in order to be able to propose a non-sinusoidal pulsation profile. This will be presented in the next section.

3.2 Column description

The disc and doughnut column that has been used is composed of a vertical cylindrical body which contains a packing of discs and doughnuts arranged and maintained by braces of constant length. Figure 4 shows a schematic presentation of such a column. The active part of the pilot column is 3.5 m and 54 mm in diameter. It consists of discs and doughnuts compartments with a 25 mm distance between disc and doughnut. A disc is 25 mm in diameter and the aperture of a doughnut is 14 mm. The open free area is subsequently nearly equal to 23 %. Liquid and solid are flowing cocurrently and the pulsation is ensured by a pneumatic system.

Andrographis paniculata is fed continuously along with the ethanol solution at the top of the column and the solution is removed continuously from the column. Consequently, the phases are forced to flow co-currently. This way of functioning is linked to the low density difference between both phases, which is not sufficient to allow a counter-current operation. Besides, in this study, the batch extraction has shown that there is no solubility limit to the transfer in the operating domain [14] and then that there is no practically need to perform a multi-stage counter-current operation.

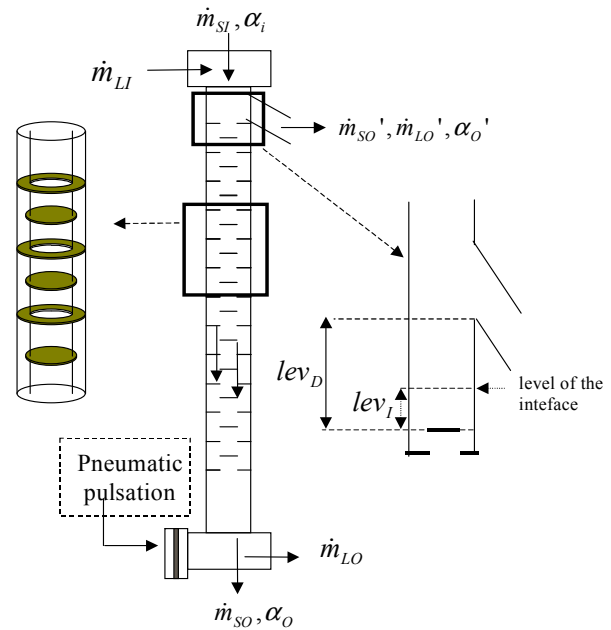


Figure 4 : Discs and doughnuts pulsed column in co-current flow with the pneumatic pulsation

3.3 Pulsation system description

An air-pulsing technique was chosen to allow variation in the pulsation policy. Figure 5 illustrates the pressure and signal (analogic) connections for the pulsation system. It is composed of a three-way valve linked in one hand to a storage tank which insures a constant 2 bars pressure and in the other hand to the room (1 bar pressure). The delivery pressure is then connected to the pulsed leg of the column and the active range is 1 to 2 bars.

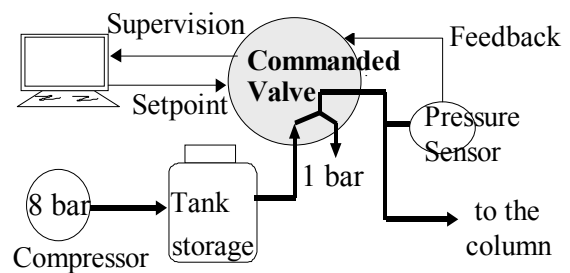


Figure 5 : Commanded valve connections for the pulsation system

A computer is used for supervision and to deliver the setpoint (in pressure) to the PID regulator. The feedback is assured by an external pressure sensor situated in the pulsed leg.

Despite its pulsing energy limits (in terms of amplitude and frequency), the proposed system gives the opportunity to have a non-sinusoidal form of pulsation. The movement of the liquid is separated into two steps :

- A pulsation step : a classic sinusoidal pulsation is used to mix the two phases in the active part of the column. This pulsation also tends to increase the solid residence time by mixing the volumes contained between a disc and a doughnut [7]. This pulsation is characterized by its frequency and amplitude f_m and A_m ;
- An impulsion step : this second step allows for a brief rise of the interface inside the column in order to purge the floating solid through the top outlet tube. This impulsion is characterised by its frequency and amplitude f_i and A_i .

Figure 6 shows the recording of a typical overall period pulsation in the experimental device.

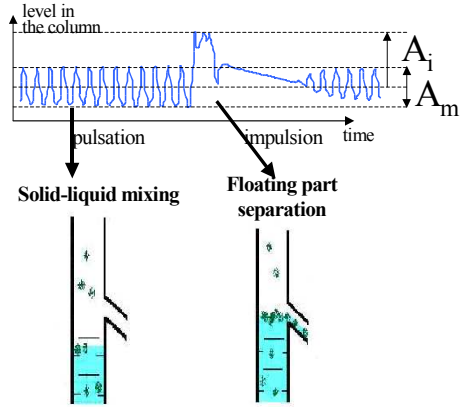


Figure 6 : Experimental pressure profile and pulsation periods

In order to minimize the energy and air consumption, it has been decided to operate at the natural frequency of the column [4, 15]. On this column, this corresponds to a frequency of 1.67 s^{-1} .

3.4 Experiments and results

The hydrodynamic behavior of the solid and liquid phases is important to be characterized in order to reach the following objectives :

- Minimizing solvent loss at the top outlet ;

- Maximizing the part of the solid going through the column (for which the extraction occurs) ;
- Obtaining a good separation between floating and sinking particles ;
- Avoiding flooding.

Three main values allow to describe these objectives :

- $\frac{\dot{m}_{SO}}{\dot{m}_{SI}}$ which is the ratio of the solid mass flow outlet at the bottom of the column over the solid mass flow inlet ;
- $\frac{\dot{m}'_{SO(sinking)}}{\dot{m}'_{SO}}$ which is the part of sinking particles among the total mass of particles going out at the top outlet;
- \dot{m}'_{LO} which is the liquid mass flow outlet at the top of column.

If $\frac{\dot{m}'_{SO(sinking)}}{\dot{m}'_{SO}}$ tends to 0, it means that the

outlet is only composed of floating particles, separation between the floating and sinking particles being assumed perfect. The maximum value of $\frac{\dot{m}'_{SO(sinking)}}{\dot{m}'_{SO}}$ should be about 0.8 because this represents the ratio between leaves and stems in the sample, that is to say between floating and sinking particles.

The last parameter \dot{m}'_{LO} can be predicted in function of the operating parameters : the liquid mass going out at the top of the column per impulsion (M_{LO}') can be described by the next equation (1):

$$M_{LO}' = \pi \frac{D^2}{4} (A_i - (lev_D - lev_i)) d \quad (1)$$

where D is the diameter of the column;

lev_D the distance between the first disk and the top outlet (figure 4);

lev_i the distance between the first disk and the interface (figure 4);

d the liquid density.

The liquid flow is then calculated according to the following equation :

$$\dot{m}'_{LO} = M_{LO}' f_i \quad (2)$$

Equation (1) is valid if there is no significant direct short-cut between the liquid inlet and the

top outlet of the column and if the level of the interface does not go over the top outlet limit during the mixing period of the pulsation (i.e. $A_m/2$ must be inferior to $(lev_D - lev_I)$).

The operating parameters quoted below are followed through experiments operated in the domain detailed in Tab.1 :

- interface level ;
- solid mass at the inlet ;
- percentage of ethanol in the solvent ;
- frequency and amplitude of impulsion.
- frequency and amplitude of pulsation

Exp. N°	Ethanol %	lev _i cm	f _m min ⁻¹	A _m psi	f _i min ⁻¹	A _i psi	m _{si} g.min ⁻¹	m _{Li} g.min ⁻¹
1	95	0.0	50.42	25.0	1.58	33.5	11.33	431.60
2	95	0.0	52.63	25.0	1.58	33.5	11.33	431.60
3	95	0.0	52.63	25.0	1.58	33.5	17.57	431.60
4	95	0.0	60.00	21.0	2.33	33.5	25.85	452.35
5	95	1.5	52.17	21.0	1.57	22	11.33	419.15
6	95	1.5	52.17	21.0	1.57	22	17.57	431.60
7	70	1.5	52.63	23.5	1.58	22	11.33	368.28
8	70	3.0	52.17	21.0	1.57	22	17.57	547.35
9	50	3.5	52.17	21.0	1.57	22	11.33	501.40
10	50	3.5	52.17	21.0	1.57	22	11.33	501.40
11	50	3.5	52.17	21.0	1.57	22	11.33	482.54
12	30	3.5	52.17	21.0	1.57	22	11.33	456.01
13	30	3.5	52.17	21.0	1.57	22	11.33	463.18
14	70	1.5	52.17	21.0	1.57	22	11.33	431.60
15	70	2.0	52.17	21.0	1.57	22	11.33	431.60

Table 1 : Operating conditions

For the whole experiments, column was operated as follows : the inlet liquid flow is started until the column is filled up with solvent and the level of the interface is then adjusted to a desired value. In a second time, the pulsation and the inlet solid flow are started.

3.4.1 Optimisation of operating parameters

Interface level

First, the liquid outlet as a function of the impulsion was studied by changing the initial level of the liquid in the column. The objective was to find the proper level of liquid in the column before starting the solid inlet. During the experiment, it is necessary that solvent losses are minimized while purging the solid. Figure 7 shows the experimental and calculated \dot{m}'_{Lo} (via equations (1) and (2)) versus the liquid level in the column. Amplitude of the impulsion is 5.5 cm, amplitude of pulsation is around 0.5 cm and

the distance between the first disc and the Tee (lev_D) is 5 cm. The good agreement allows to validate the equation (1) and the hypothesis of the calculation. As expected, it can be observed on figure 7 that the higher the level of the interface, the more important is the volume going over the outlet limit. So, the appropriate level of the liquid should be close to the first disk.

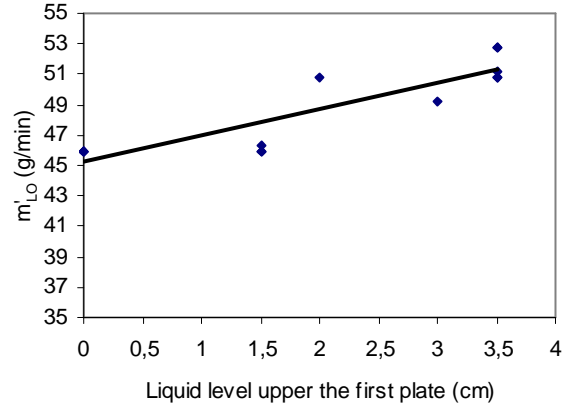


Figure 7 : Mass liquid outlet as a function of the initial liquid level in the column

The influence of the interface position on the separation efficiency has been also studied and the results are presented on figure 8. Remember that the optimal efficiency is expected when $\frac{\dot{m}'_{SO(sinking)}}{\dot{m}'_{SO}}$ tends to zero,

whilst the worse condition coincide with a value equal to the initial ratio leaves-to-stems of 0.8. First, we can notice that $\frac{\dot{m}'_{SO(sinking)}}{\dot{m}'_{SO}}$ is roughly comprised between 0.2 and 0.3 and seems to be almost constant according to the interface level. If one wants to be more accurate, it is shown that there is a slight increase with increasing liquid level. The reason for that is unclear, probably linked to the accumulation of solid on the disc. When the interface level is close to the disc, there is a better solvent impregnation of the solid phase due to this natural accumulation. On the contrary, when the interface level is close to the Tee junction, the fresh solid is not as well wetted as it was close to the disc. However, whatever the reason is, it is recommended to operate in the vicinity of the first disc.

Frequency and Amplitude of impulsion

At the top of the column, the solid in contact with the liquid absorbs the solvent. As explained in paragraph 3.1, even after a long

time of contact (over 5 hours), parts of the solid are still floating. The time of contact depends on f_i . It must be long enough to obtain a majority of sinking particles around the interface level which will go through the column instead of being evacuated after the impulsion. Experiments have been performed with one impulsion every 38 min.

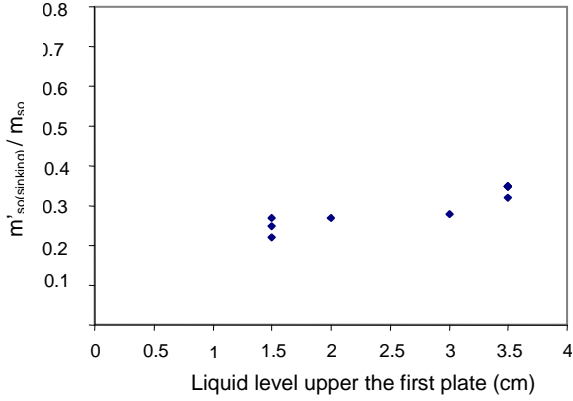


Figure 8 : Ratio of sinking solid going out at the top of the column as a function of the liquid level upper the first plate

According to equation (1), $A_i/2$ must be superior to $lev_D - lev_l$ to purge a minimum of solid. A_i must be tuned to eliminate the exact height of floating solid ($\Delta h_{floating}$) :

$$\frac{A_i}{2} = lev_D - lev_l + \Delta h_{floating} \quad (3)$$

Frequency and Amplitude of pulsation

This work does not concentrate on the analysis of the influence of the frequency and the amplitude during the mixing period of the pulsation (sinusoidal part). The only objective is that f_m and A_m permit to reach a sufficient level of mixing in the column. Only A_m is subjected to the constraint stated above :

$$\frac{A_m}{2} < lev_D - lev_l \quad (4)$$

Solid Mass inlet

Figure 9 shows the ratio $\frac{\dot{m}_{SO}}{\dot{m}_{SI}}$ in function of the mass solid inlet at 95 % ethanol. $\frac{\dot{m}_{SO}}{\dot{m}_{SI}}$ decreases and, reciprocally, the proportion of solid at the top outlet $\frac{\dot{m}'_{SO}}{\dot{m}_{SI}}$ increases when the mass solid inlet increases.

Besides, the separation is more efficient for a small flow of solid at the inlet, like can be seen on figure 10. This can be explained by the fact that some solid does not reach the liquid and remains dry if the solid flow is too high. This confirms that solid particles sink more easily when a good contact has been established between the two phases.

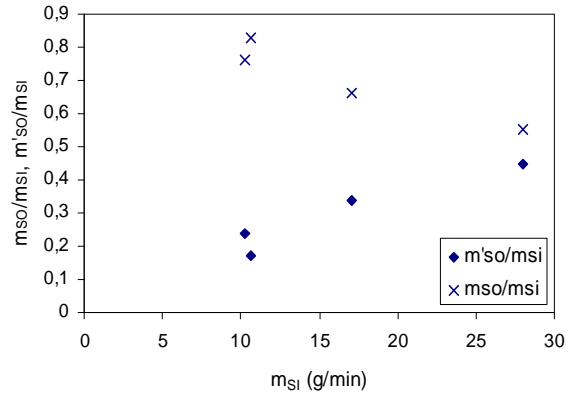


Figure 9 : Solid flow rate ratio at the bottom and at the top of the column as a function of the mass solid inlet

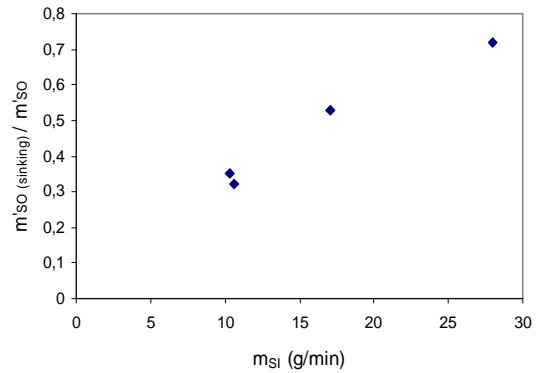


Figure 10 : Ratio of sinking solid going out at the top of the column as a function of the mass solid inlet

Percentage of ethanol

Experiments allowed to emphasize the importance of the percentage of ethanol. As illustrated in figures 11 and 12, when the percentage of ethanol increases, \dot{m}_{SO} increases while α_0' decreases. As kinetics of solvent absorption is enhanced by high percentage of ethanol (figure 3), particles sink in greater quantity and faster.

During the experiments the behavior of solid particles was observed as a function of percentage ethanol :

- For a percentage of ethanol around 95%, the sinking particles only consist of leaves.
- For a percentage of ethanol much smaller than 95 %, some leaves are present in the floating part.

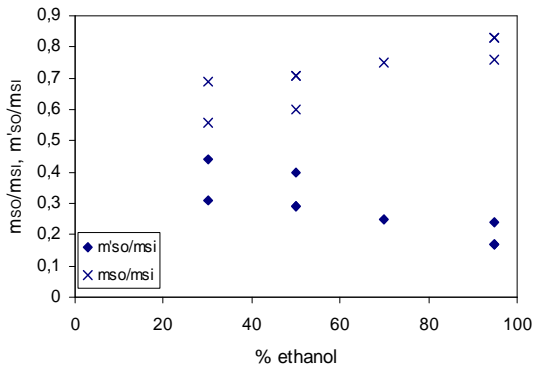


Figure 11 : Solid flow rate ratio at the bottom and at the top of the column as a function of the percentage of ethanol

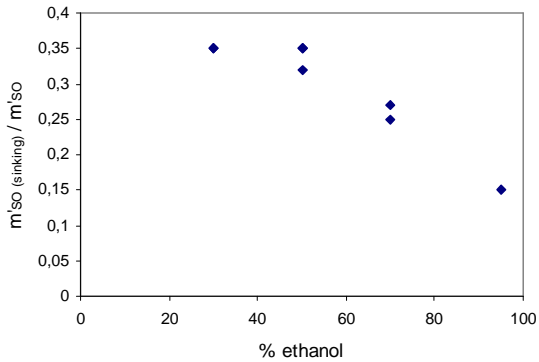


Figure 12 : Ratio of sinking solid going out at the top of the column as a function of the percentage of ethanol

3.4.2 Experimental operated domain,

The operating points have been reported on figure 13. The limit of flooding point in function of the mass solid inlet and the percentage of ethanol is also represented, whereas no operating condition could be determined when developing this extraction in a classical column with a simple sinusoidal due to floating particles. It is shown that this problem has been solved with the recommended method.

3.4.3 Conclusion on continuous extraction and proposal for a design

The air pulsing technique allowed changing the regular sinusoidal agitation. The hydrodynamic behavior of the particles strongly depends on the ethanol concentration and on the mass flow inlet. But, for a large range of operating conditions, the separation of the floating and sinking solids at the top of the column thanks to pulsation produced encouraging results. This example proves that the pulsation is an

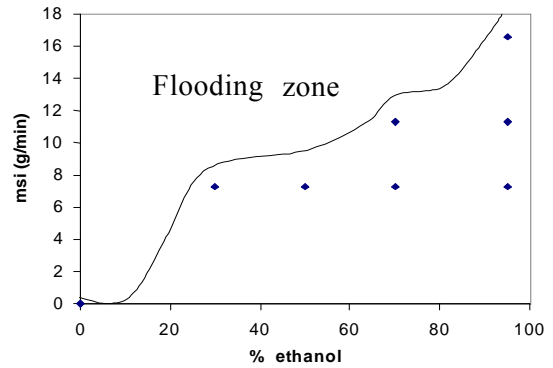


Figure 13 : Experimental operated domain and flooding zone function of the percentage of ethanol and the solid flow rate

interesting tool to transpose batch operations involving solid phase to continuous processes. An extraction yield around 30% for a 4 min residence time in the 2.5 m column was obtained. This yield has to be compared with the batch experiments which showed a similar efficiency for the same contact time. To reach larger residence times, changing the intensity of the pulsation (amplitude or frequency) can be a useful tool, but not enough powerful, especially for dense particles which have a fast settling velocity. Otherwise, the column height should be increased.

For an industrial application, two main technological modifications can be suggested to improve the separation of particles. The first concerns the upper part of the column. Its diameter can be increased in order to avoid the clogging of floating solid while purged by the impulsion. Enlarging the diameter beneath the solid inlet is also a way of extending the range of solid mass flow inlet. With a wider interface section, the contact surface between the solid feed in and the column liquid will be increased and less solid will remain dry. Finally, in order to obtain a better extraction yield, the set up of

the solid inlet at an intermediate position in the column would allow to extract the solute present on the floating particles mainly composed of stems. The floating particles could rise while liquid phase would go down during the pulsation step. During the impulsion, the floating solid having reached the top of the column would be removed. This new position of the inlet would also prevent any limitation on mass solid flow inlet, whereas the mixing of both phases is optimised.

4 Conclusion

With the review presented at the beginning of this article, it could be noticed that the potentiality and the flexibility of various pulsation systems existing was not fully exploited. Technological innovations using pulsation are often specific to a particular case, to solve a problem met during operation. And, they are not generally completed by a more theoretical study which would provide design equations. Here, we proposed a more systematic methodology which consists in adapting the shape of the pulsation derived from a sinusoidal signal, to the problem of transport encountered. The reciprocation of a pulsation step and an impulsion step successfully allowed, in andrographolide example, to avoid flooding. Given that the modern trend of chemical engineering is to head for "Process Intensification", innovations in pulsation policy is therefore justifiable in seeking to increase residence time of solids in continuous columns.

Nomenclatures

A	Amplitude of pulse (cm)
A_i	Amplitude of impulsion (cm)
A_m	Amplitude of mixing (cm)
d	Liquid density (g/cm^3)
D	Diameter of the column (cm)
f	Frequency of pulse (sec^{-1})
f_i	Frequencies of impulsion (sec^{-1})
f_m	Frequencies of mixing (sec^{-1})
k_1, k_2, k_3	Constants
lev_D	Distance between the first disk and the top outlet (cm)
lev_I	Distance between the first disk and the interface (cm)

\dot{m}_{LI}	Mass liquid flow inlet (g/min)
\dot{m}_{LO}'	Mass liquid flow outlet at the top of column (g/min)
M_{LO}'	Liquid mass going out at top column per impulsion (g)
\dot{m}_{LO}	Mass liquid flow outlet at the bottom of column (g/min)
\dot{m}_{SO}'	Mass solid flow outlet at top column (g/min)
\dot{m}_{SO}	Mass solid flow outlet at bottom column (g/min)
m_{SO}'	Mass solid outlet at top column (g)
m_{SO}	Mass solid outlet at bottom column (g)
\dot{m}_{SI}	Mass solid flow inlet (g/min)
P	Pressure of the air reservoir (bars)

Greek letters

α_0	Sinking mass outlet / total solid mass outlet (at bottom of column)
α_0'	Sinking mass outlet / total solid mass outlet (at top of column)
α_i	Ratio of sinking mass and total solid mass in the sample
$\Delta h_{floating}$	Height of floating solid (cm)

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