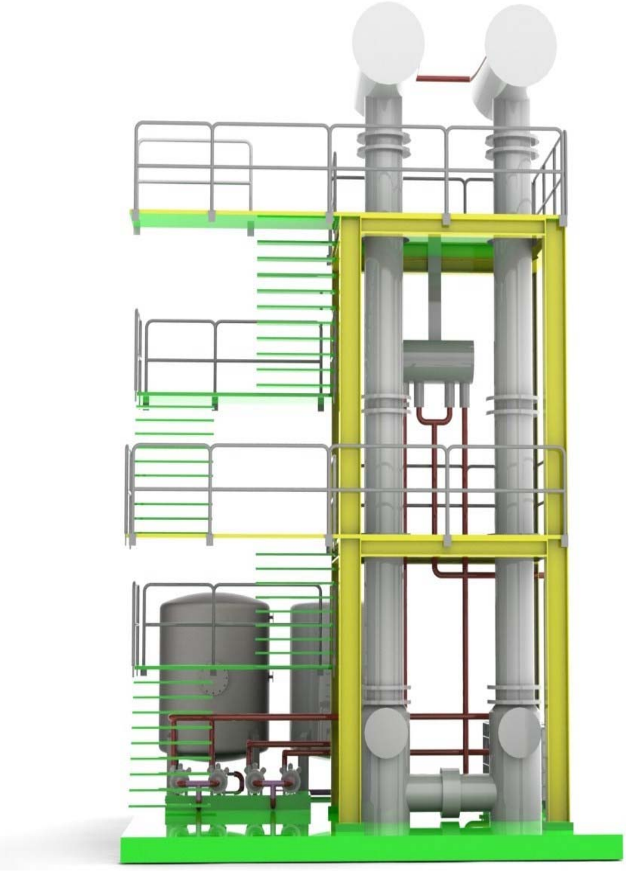
PULSED U-COLUMN – SIEVE PLATE TWIN COLUMNS



The pulsed U-column consists of two normal, continuous working, pulsed columns, arranged as a U-tube. The columns are identical and each consists of:

- A vertical, cylindrical tube containing a packing of horizontal, perforated plates
- Two larger horizontal tubes at the top and at the bottom to allow phase disengagement
- A pulsatory device, a pneumatic operated membrane, in between the two columns that periodically moves the liquids inside the columns up and down.

The twin column arrangement has the advantage that the pulses are swinging like a gyratory movement between the two columns, giving low energy input and a smooth operation, no heavy lifting of liquid pillars. Both columns don’t need to have the same function, e.g. one can be designed for extraction, the other for stripping.

General Description of a Pulsed Column

A pulsed sieve-plate extraction column is an often-used industrial equipment, because of its considerable flexibility, high throughput and high separation efficiency. Another advantage of the column is there insensitivity to contamination. On the other hand, there are factors, e.g. the drop coalescence, the ability of the used liquids to wet the plate surfaces and the effect of impurities that are poorly understood and that limit the use of theoretical approaches. Therefore, pilot plant tests may be necessary to design a column.

The column consists of a vertical, cylindrical tube with a number of stationary, horizontal, perforated plates at equal distance from each other. The plates have a certain number of holes of a specific size. Two immiscible liquids with different densities are added to the column. The liquid with the lowest density is added from the lower part of the column and is forced upwards. The liquid with the higher density is added from the top and is falling down the column by gravity. When the liquids get in touch with each other a counter current flow is created. Because the two liquids are immiscible, dispersion is formed that creates drops in the continuous phase. When the liquids reach the opposite end of the column from which they entered, the phases are coalescing into two unmixed liquids. The heavier phase is leaving the column from the bottom through a jackleg, and the lighter phase is leaving the column from the top through a weir. The position of the interfacial boundary is decided by the continuous phase.

In the column, there is a device generating pulses. The pulses bring about regular up- and downward liquid movements. The liquids (phases) are alternately forced through the holes of the plates. The lighter phase is rising in the upward pulse, and the heavier phase is falling in the downward pulse. The pulsation is sinusoidal and the product of the frequency (**f**, pulses per unit of time), and the amplitude (**a**, the piston stroke length) and makes the liquids move through the column. Without the pulsation, no counter current flow is possible due to capillary effects.
The use of pulsed columns is limited by the physical properties of the liquids and too large liquid flows. The efficiency of the pulsed column also depends on a number of other factors; the pulsation, the flows, the phase ratio, which phase is dispersed and which is continuous, plate wettability, the geometry of the plates, the concentration of the added component, drop coalescence, the diameter of the column and the effect of impurities. The separation effect of the column decreases with increased diameter.

The size of the drops is a very important parameter for the design of a pulsed column. It affects the residence time of the dispersed phase, and the flooding behaviour of the column. Furthermore, it affects the interfacial area, which is available for mass transport. At low pulsations, the drops are large, and the dispersed phase can only pass through the plates due to the pulse. Because of that, the dispersed phase can coalescence under the plates between each pulse, which makes the column act like a number of mixer-settlers. At higher pulsations, the drops are small enough to pass freely between the plates. The liquids in the column are then flowing counter currently.

The drop size distribution depends on the interaction between drop decomposition and drop coalescence. The drop size gets smaller and the drop size distribution gets narrower with increased pulsation. That means a larger area where mass transport can occur. Almost the whole reduction in mean drop diameter is happening during the passage through the first plate. After a certain number of plates the drop size distribution is stable. Where this occurs, depends on the plate geometry, interfacial tension of the liquid system and pulsation. Thus, the drop size distribution is independent of the column diameter, flows and phase ratio.

Mass transfer

Mass transfer is the transfer of the dissolved component from one phase to another, physically separated from the first one. The mass transfer is the basis for separation by solvent extraction. When deciding which phase to disperse, the direction of mass transfer must be taken into account. The best separation efficiency is obtained when the direction is from the continuous phase to the dispersed phase. The interfacial tension and hence the drop size is reduced by the component being transferred, leading to a lowering of the column throughput.

Hold-up, that is the volume fraction of the dispersed phase in the plate part of the column, is just like the drop size distribution an important parameter for describing the mass transfer area of the column. The hold-up is directly affected by the throughput of both phases, and by the relative velocity of the drop swarm. The relative velocity is, in turn, a function of the physical properties of the liquid system, the drop size, and the retarding effects of pulsation and sieve plates on the drop motion.

Flooding and the Flooding Curve

Flooding means that the liquids leave the column from the same end as they are entering. It is often a sign of too high flows. Flooding occurs when the liquids act as hydraulic hindrances and block each other from flowing counter currently. Flooding can occur both above and below the region of stable operation. Flooding because of too low pulsation will occur when insufficient mechanical agitation is supplied to force the liquids through the holes of the plates. In this case, the plates work as a physical flow barrier.

When the column is flooding, it does not work in a satisfactory way. The diameter of a satisfactory working column is thus dictated by the flooding throughput. Therefore, information on flooding characteristics is important for the column design.

The characteristics of the operation of a pulsed column are well documented in the literature and commonly described with the Sege & Woodfield diagram shown below.

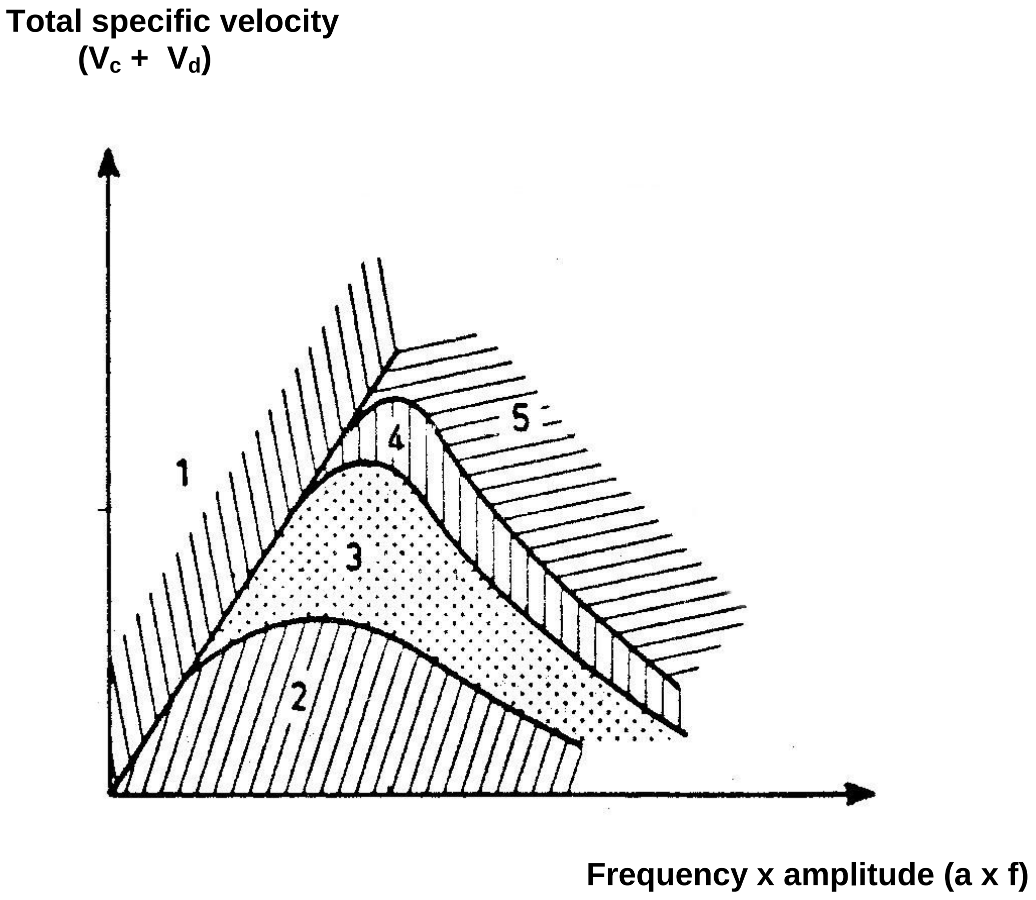


Diagram: Sege & Woodfield Diagram

1. Flooding region
2. Mixer-settler operating region
3. Emulsion operating region
4. Instable operation region
5. Flooding region

$$(V_c + V_d) = (Q_c + Q_d)/A; \ \mathrm{m/s}$$

V is flow velocity, Q is flow rate and A column area

$$a \times f ; \ \mathrm{m/s}$$

a is amplitude, f is frequency

A flooding curve is a diagram describing the maximum column throughput. The total flow velocity (y-axis) is plotted against the pulsation (x-axis). The total flow velocity is the sum of the continuous and the dispersed flow per unit of time and cross section area. The pulsation is the number of pulses per unit of time, multiplied by the pulse volume displacement and divided by the cross section area.

The highest total flow velocity at each pulsation where the column does not flood is indicated in the diagram. When a line is drawn between the points a flooding curve is created. The shapes of different flooding curves are usually similar. At low pulsation the capacity of the column equals the pulse volume velocity and is therefore increasing proportional to the pulsation. This relation forms a straight line from the origin towards the upper right part of the diagram. When a maximum has passed the capacity decreases with increased pulsation until no counter current flow through the column is possible.

The regions are the *mixer-settler region* (1), the *emulsion region* (2), an *unstable region* (3), *flooding* due to insufficient pulsation (4), and *flooding* due to too large flows (5). The changes in dispersion behaviour involve gradual blending of one type of operation to another rather than abrupt transitions.

The *mixer-settler region occurs* at low throughput and low pulsation. It is characterized by separation of the lighter phase in a discrete and clear layer beneath the plates and between each pulse. The mixer-settler region is a stable region, but the mass transfer is ineffective compared to the emulsion region.

The *emulsion region occurs* at higher pulsations and throughput rates. This region is characterized by small drop sizes and homogeneous dispersion. This results in a large contact area between the phases, creating a good mass transfer. In the emulsion region, the dispersion is maintained during the whole pulse, which is not the case in the mixer settler region. Also, the drops are sufficiently small to pass freely through the plate perforations.

The *unstable region* occurs at even higher throughput rates and pulsations. It is characterized by a mixture of small and larger drops of the dispersed phase in the continuous phase, drops of irregular form and local flooding. The efficiency of the column is often lower and varies more than in the emulsion region. When the throughput rates or the pulsation is increased even more, total flooding eventually occurs.

As described above, the emulsion region is the desirable working region. Because a large flow means that a greater amount can be separated in a shorter time, an as large flow as possible is desirable. For these reasons, the form of the curve, which corresponds to the maximum of the emulsion region, is demanded.

Pulsed Column Design and Empirical Correlations

Unfortunately, there are still many uncertainties related to purely theoretical approaches to design of pulsed columns. This is among other things due to the complex relationship between column behaviour and operating parameters. Other factors poorly understood are the drop coalescence mechanisms, plate wettability and effect of impurities. It is the common opinion that reliable dimensioning of pulsed columns is only can be based on results of pilot plant tests with the actual liquid system. Such experiments are time consuming and require large amounts of liquids. In order to substitute these tests with laboratory-scale experiments, there have been needs for theoretical models that can be used to scale up the diameter of the column, and to adjust it to the optimal flow. According to the literature, there is no general empirical model that can describe flooding in a very accurate way by using all column parameters. This is also true for certain liquid systems without mass transport.

Several empirical correlations with the purpose to be used for approximate calculations of the maximum volume velocity in a flooding curve are found in the literature. Some of them can be used to calculate the maximum point of the flooding curve, while some of them can be seen as a whole curve at different pulsations when they are plotted. One each of the two types of correlations is mentioned below.

The following expression is of the first type, calculating the maximum point of the flooding curve:

$$(\mathbf{V_C} + \mathbf{V_D})_{\max} =$$

$$(24{,}528 + 2{,}537\,\gamma - 0{,}0548\,\gamma^2)(1 - 1{,}455\,\varepsilon + 3{,}247\,\varepsilon^2)(1 + 0{,}1778 \ln \mathbf{V_C/V_D} + 0{,}0437[\ln \mathbf{V_C/V_D}]^2) \qquad (1)$$

where $\mathbf{V_C}$ is the continuous flow per unit of time and cross-section area [m/h], $\mathbf{V_D}$ is the disperse flow per unit of time and cross-section area [m/h], γ is the interfacial tension [10^{-3} N/m] and ε is the free fractional plate area.

The following expression is also suggest using the correlation for determining the maximum point of the pulsation frequency:

$$\mathbf{f_{max}} = 29{,}450 + 6{,}679\,\gamma - 0{,}1082\,\gamma^2 - (2{,}067 + 0{,}426\,\gamma) \ln \mathbf{V_D/V_C} \qquad (2)$$

The symbols are according to expression (1). Experience indicates that the pulsation, resulting in near optimum performance will lie somewhere in the range from 75 to 95 % of the flooding pulsation.

These correlations are applicable to liquid systems of high to moderate interfacial tensions and to plates of small to moderate free areas.

When calculating the optimal frequency according to equation (2) the amplitude is not a parameter. Variation of the amplitude has effects not fully accounted for by the corresponding amplitude-frequency product. When a certain size of plate spacing is used, certain amplitude is optimal in addition to a certain amplitude-frequency product. Furthermore, flooding and good emulsion type operation occurs at different amplitude-frequency products depending on the amplitude.

Other types of correlations are suggested in the literature to describe flooding curves. The different correlations often have very similar appearances.

When the total flow velocity has been predicted, the requisite column diameter (D) can be calculated by using equation 3:

$$D = [4(Q_C + Q_D)/\pi\ F(V_C + V_D)]^{1/2} \qquad (3)$$

where F is the fraction of the flooding velocity at which the column is to operate and the remaining symbols are defined in the diagram above.

Comments

The opinions regarding the throughput flow rates dependency of the column diameter are divergent. Some state that the throughput rates and the pulsations for stable operation are not appreciably affected by a small scale-up of the column diameter. However, it has been noted that a small column sometimes undergoes flooding more readily than large columns. In a highly unstable operation, a globe of coalesced dispersed phase may fill the entire cross section of the small column. The globe obstructs the counter current flow sufficiently to cause flooding. In the large column a globe of similar size will not fill the entire cross section.

Choosing a larger diameter with the lower flow rate will not necessarily be a preferred alternative because a diameter decrease gained from a column design based on a high flow rate often must be compensated for with increased column height.

The differences between empirical correlations can be large. The best possible correlation should of course agree with the performance of the U-type pulsed column. However, there are other important properties as well. Some equations are much more complicated and has a great amount of varying parameters.

It has been noticed that sources in literature using the more simple equation (1) are several decades younger than the ones using more complicated equations. The reason why the simple equation with less parameters are more frequently used can be that interfacial tension has the greatest effect on the fluid dynamics of an extraction column, which makes the other parameters less important.