

EXPERIMENTAL STUDY FOR FLOODING CHARACTERISTICS IN PULSED SIEVE EXTRACTION COLUMN

Submitted in partial fulfilment of the requirements for the award of
Bachelor of Technology Degree in Chemical Engineering

By

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SCHOOL OF BIO & CHEMICAL ENGINEERING**

SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

Accredited with Grade “ A ” by NAAC

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This is certify that this Project Report is the bonafide work of **SANDEEP.O.S (Reg No 39690016)** and **RAUDA SRIKRISHNA (Reg No 39690023)** who carried out the project entitled “ **EXPERIMENTAL STUDY FOR FLOODING CHARECTERISTICS IN PULSED SIEVE EXTRACTION COLUMN** ” under our supervision from July to October.

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ABSTRACT

The methodical investigation of pulse column operational factors, such as air reservoir pressure, pulse amplitude, pulse frequency, light liquid flow rate, and heavy liquid flow rate, which affect flooding.. The experimental study was made of flooding in a 50mm. diameter and 40 number of plates pulse column, using a water-normal paraffin hydrocarbon system under no mass transfer conditions. An analysis of column operation led to functional relationship between column operating parameters such as pulse frequency, pulse amplitude and air reservoir pressure. A quick, easy method was created to identify floods brought on by excessive and insufficient pulsation. The flooding characteristics curve was determined by experimentally kept pulse amplitude made as constant.

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LIST OF SYMBOLS

Symbol	Description
a	amplitude of the pulse
f	frequency of the pulse
K	proportionality constant
F	volumetric flow rate
V	volume of contactor
ε	dispersed phase holdup
U_{slip}	slip velocity
U_d	dispersed phase velocity
V_k	characteristic velocity
HTU	height transfer unit

1. INTRODUCTION

1.1. LIQUID-LIQUID EXTRACTION

By contacting a liquid mixture with another liquid, the mass transfer process known as liquid-liquid extraction (LLX) is used to separate the components of a liquid combination. There could be a wide variety of components in the feed stream. It must consist of at least two parts. Typically, we only want to extract one component from a feed stream. This part is referred to as a "Solute." Carrier liquid is the collective name for the elements that aren't the solute. Typically, the solvent stream is a clear liquid. The solvent is chosen in such a way that it should only dissolve the solute component—the component we are interested in—and not other components. In the event that the solvent dissolves all of the components, the solvent used should dissolve more of the component of interest than the other components. When they come into contact, depending on how much feed stream or solvent stream is being utilised, it often happens that one of the liquids disperses into the other liquid in the form of tiny droplets. The liquids' surface tension should be such that it prevents minuscule droplets from moving into the other phase during mixing but must maintain the liquids apart once mixing is complete. The rate of mass transfer of the solute from the feed to the solvent increases with the size of the droplets. If the density difference between the two liquids is insufficient, the two phases will not easily or instantly separate after mixing and will instead exist as a dispersion, which is known as an emulsion.

The Extraction Process Feed Solvent Raffinate Extract temperature of the process can be raised, or another element that aids in the separation of the phases can be added, to break the emulsion. These parameters also affect the density of liquids. If this is not done, we will need expensive, space- consuming, and very massive extraction equipment, which may render the entire procedure impractical. After mixing is complete, the mixture separates into two phases; the extract phase, which is ideally composed of both solute and solvent, is the phase that is rich in solutes. The Raffinate phase, also known as the solute-lean phase, consists primarily of the carrier liquid and a small amount of solute. If the procedure is not optimal, a small amount of solvent and carrier liquid may also be present in the raffinate phase and extract phase, respectively.

Extraction might be a method of preference when:

- If the components that need to be separated have close boiling points or are heat sensitive, distillation cannot be utilised to separate them.
- The feed itself contains very little solute; it is essentially a diluted liquid mixture.
- In the metal extraction industries, we must distinguish between non-volatile metal solutes.
- The aqueous stream needs to be cleaned of organic matter.

1.2. LIQUID-LIQUID EXTRACTION EQUIPMENT'S

1.2.1. MIXER-SETTLERS

A single stage of extraction is provided by each mixer-settler unit. The two phases are combined by an impeller as they enter the mixing area. Due to the variations in their densities, the two phases of the two-phase solution flow into the settling section where they are permitted to separate by gravity. The typical mixing and settling timeframes for mixer settlers are on the order of a few minutes. The different phases either flow over a weir (organic solution) or through an underflow and then over a weir as they leave the settling section (aqueous phase). The height of the weirs on the exits of the settler portion controls the separation interface. The only mechanical maintenance needed is the occasional replacement of the mixing motor, and only minimal instrumentation is needed. Multiple mixer settlers are installed in a counter-current process, with mixing and settling chambers placed at the opposite ends of each stage (because the outlet of the settling sections feeds the inlets of the mixing sections of the following stage's settling sections). When a procedure needs longer residence times and the solutions may be easily separated by gravity, mixer-settlers are used. They need a huge facility footprint but little headroom, and they only need limited remote maintenance capabilities for replacement of mixing motors on occasion.

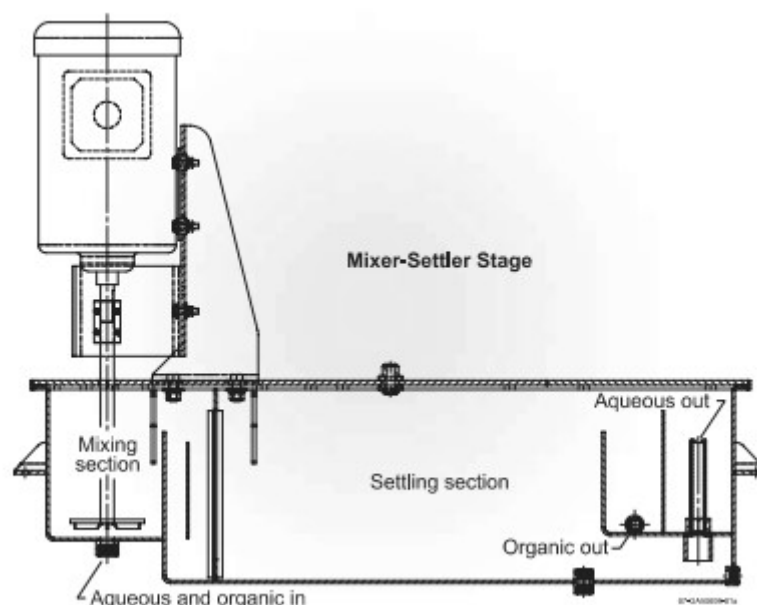


Fig: 1.1: Diagram of a mixer settler

1.2.2. CENTRIFUGAL CONTACTORS

Similar to mixer-settlers, centrifugal contactors are discrete-stage devices that offer one stage of extraction per unit. They can be easily connected as each rotor pumps separated fluids to the next stage inlet in both directions. The two-phase mixture separation is the main distinction between a centrifugal contactor and a mixer-settler. Centrifugal contactors use a spinning rotor to efficiently and quickly separate the two phases after vigorously mixing them together inside the rotor, where centrifugal forces can reach 300 g. Similar to a mixer-settler, the divided phases exit the contactor through overflow and underflow weirs. Figure 1.2 displays a cutaway view of a centrifugal contactor in use.

For chemical processes with quick kinetics, centrifugal contactors typically achieve high single stage efficiencies (more than 95% of theoretical). If centrifugal contactor rotors are kept spinning, process flow interruptions do not result in a loss of process concentration profiles. As a result, operations using centrifugal contactors can be "paused" for a length of time long enough to restart flow or even swap out a motor without suffering a major loss in output or rework. Centrifugal contactors only need a minimal amount of instrumentation to operate. Monitoring of motor amperage, rotor rpm, inlet flow rates, temperatures, and many other process parameters is possible with

computer control using commercial software. When a process requires brief residence times, on the order of several seconds, centrifugal contactors are used. They only need a small amount of floor space and headroom, but they do need remote maintenance capabilities for routine removal of the motor and/or rotor.

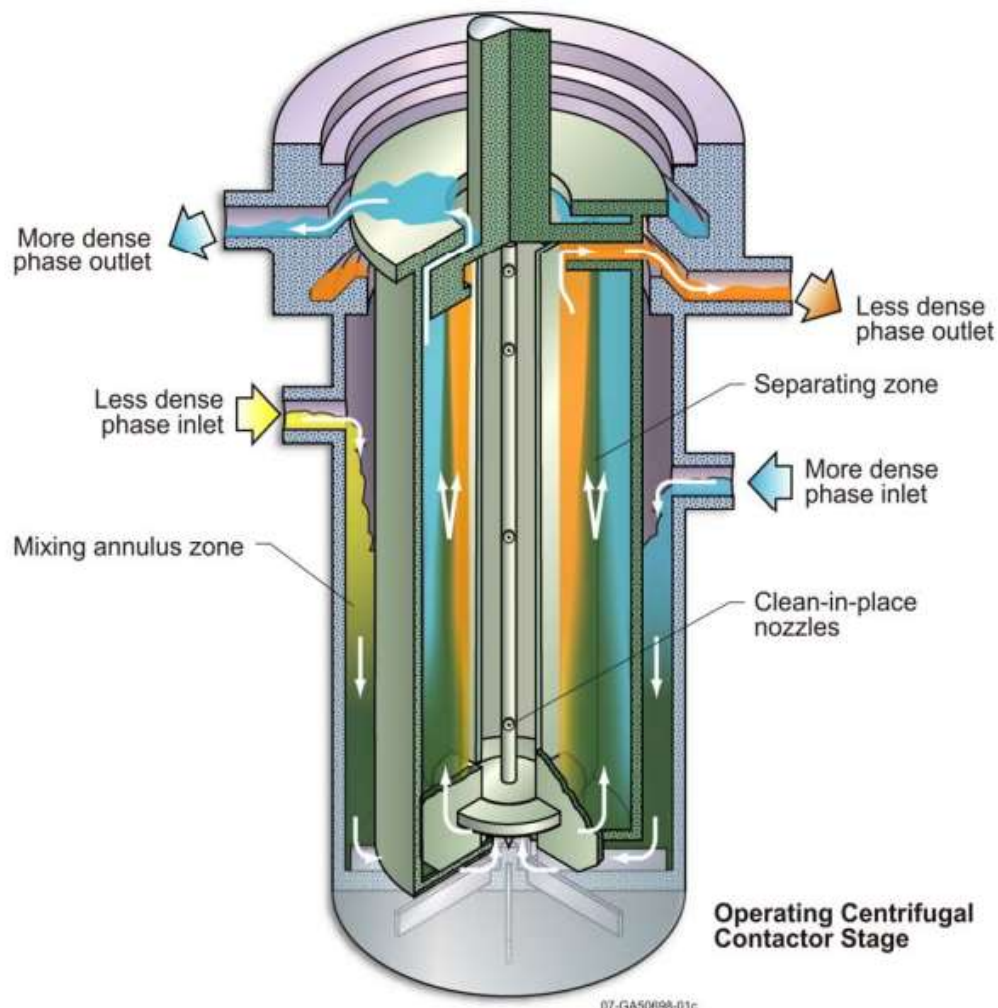


Fig: 1.2: Cutaway view of a centrifugal contactor in use

1.2.3. COLUMNS

The two most common types of columns used in industrial settings are packed columns and pulse columns with plates or trays.

Packing material, such as Ranching Rings, is added to packed columns to provide a winding path for the two solutions as they flow through the column, ensuring that

the two phases are continually in contact (usually with aqueous feed down and solvent up).

Although packed columns don't have any moving parts and are comparatively simple to use, they are not very efficient.

Because columns lack distinguishable stages like mixer-settlers or centrifugal aqueous out aqueous and organic in settling sections,

The number of stages in the mixing section's calculation is based on the height of a hypothetical stage. The number of stages in the Mixer-Settler contactors of the mixing section is determined by the height of a hypothetical stage. This height is often many feet for most packed columns, which means that very tall columns would be needed for a counter-current process using multiple stages to reach a particular separation factor.

Other packing (trays or perforated plates) is used to decrease the height of a theoretical stage in the column and mechanical energy is employed to push the dispersed phase into smaller droplets, enhancing mass transfer. The pulse column is the most often utilised type of column, particularly in the nuclear industry. As with a packed column, liquids are continually delivered into a pulse column where they flow counter-currently, but mechanical energy is used to pulse the liquids in the column up and down. In order to accomplish this, pressured air is often injected into a pulse leg, pushing liquid into the column, and then the pulse leg is vented, filling the pulse leg with solution from the column. Usually only a few inches, the solution in the column is raised and lowered by the pulse action. As the dispersed phase is forced through the perforated plates or other plates in the column, droplet formation is encouraged. This pulse decreases the dispersed phase's droplet size and enhances mass transfer. Figure 1.3 depicts a perforated plate pulse column. Many different plate designs are employed. Sieve plates, which are flat plates with holes bored into them, were employed in the earliest pulse columns. The nozzle plate, which has distinct curves on the top and bottom of the plate, is a more efficient plate (making it directional, in that it must be configured according to the continuous phase in the column). The "disc and donut" form is used by the French and Japanese pulse columns. The plates are solid (have no apertures), however the alternating plates allow for efficient phase contacting.

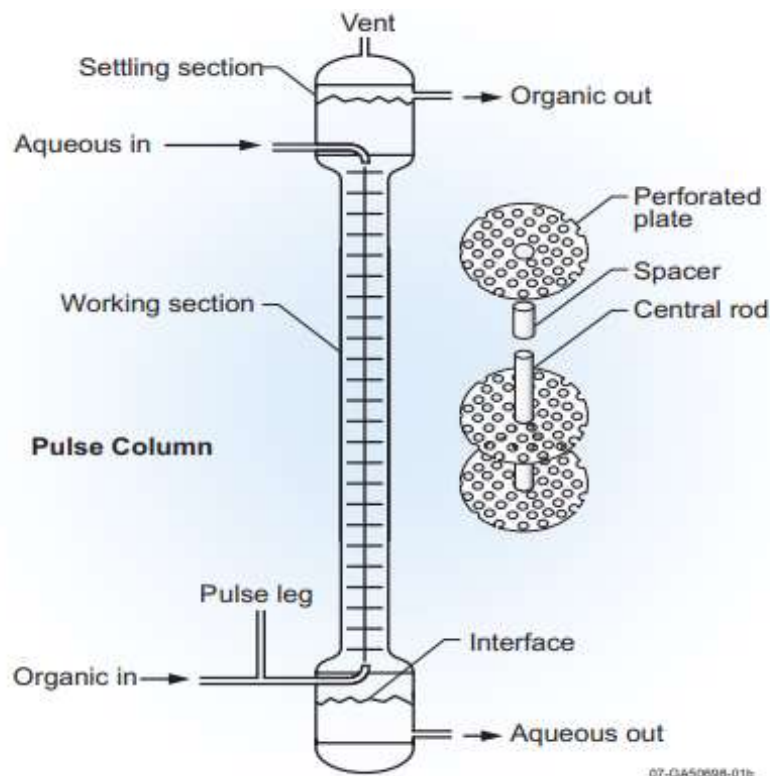


Fig: 1.3: Pulse column with perforated plate

Using bubble probes in the disengaging section, the separation interface is managed while the column is in operation. The probes are connected to a controller that removes the heavy phase from the column's base. By calculating the ratio of heavy to light phases in the column, the bubble probes enable operators to monitor the "weight" of the column, which provides them with a good indication of column performance. Pulse frequency and amplitude must be adjusted during operation, therefore pulsing devices and pulse speed controls are also necessary. Only the pulsating apparatus, which is out of cell, above the columns, needs periodic maintenance. When a procedure calls for intermediate residence times, pulse columns are utilised because the flow rate may be quickly changed to change the residence time. They need a modest facility footprint yet a lot of headroom (typically 40-50 feet). Due to the fact that all moving parts (pulser equipment) are situated outside of the shielded cell, pulse columns do not require remote maintenance capability.

1.3. APPLICATIONS OF LIQUID- LIQUID EXTRACTIONS

The liquid-liquid extraction method is frequently used in the nuclear industry to recover uranium and plutonium.

The extraction process is particularly suited to the petroleum industries since heat-sensitive liquid feeds must be separated based on their chemical type (for example, aliphatic, aromatic, and naphthenic) rather than their molecular weight or vapour pressure.

The separation of antibiotics and protein recovery is prioritised in the biochemical and pharmaceutical industries, where there are also considerable applications.

They are used to extract sodium hydroxide, phosphoric acid, and other high boiling compounds from aqueous solutions in the inorganic chemical sector.

2. LITERATURE SURVEY

2.1. PULSED COLUMN:

Pulsed column is a differential contactor type solvent extraction column (Liquid Liquid Contactor). Pulsed agitated columns were first patented by Van Dijck. Pulsing increases turbulence and hence mass transfer rate. Thereby, it helps in increasing the extraction efficiency. Pulsation of liquid in the column is done through a series of perforated plate are known as catridges.

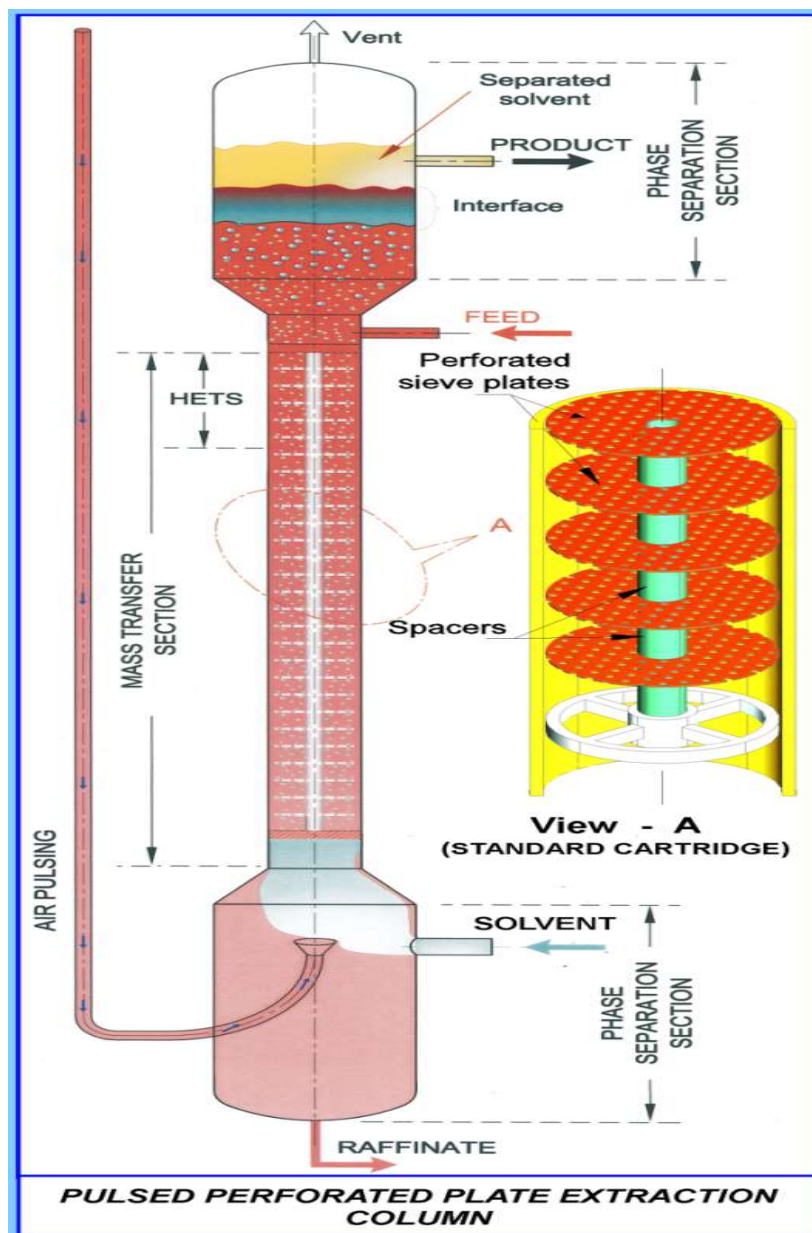


Fig: 2.1: Pulsed Perforated Plate Extraction Column

The pulse column operations can be divided into five distinct zones. These are,

- Flooding due to insufficient pulsing
- Mixer-settler region characterised by separation of phases into clear layers between plates during the quiescent portions of the pulse cycle.
- Emulsion type region, characterised by fairly uniform dispersion of the discontinuous phase and little change in the phase dispersion throughout the pulse cycle.
- Unstable region characterised by local flooding and irregular dispersion at operation neat the flooding point.
- Flooding region due to excessive pulsation.

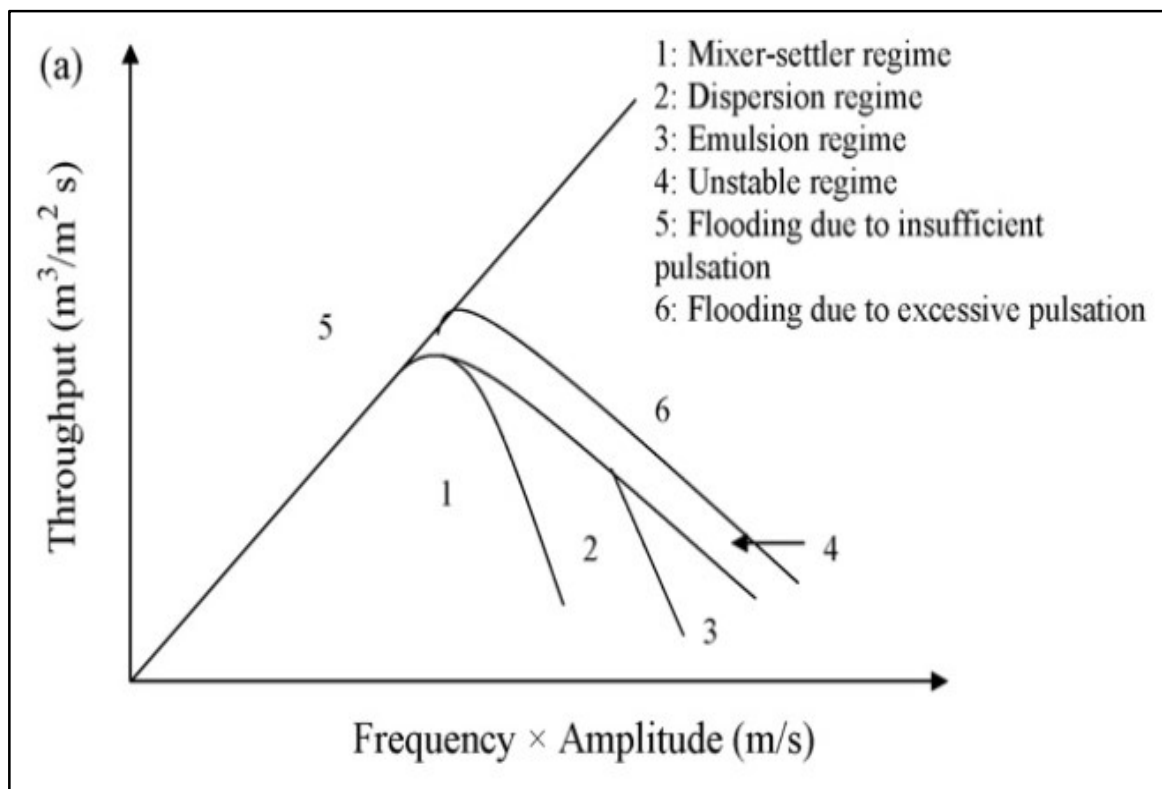
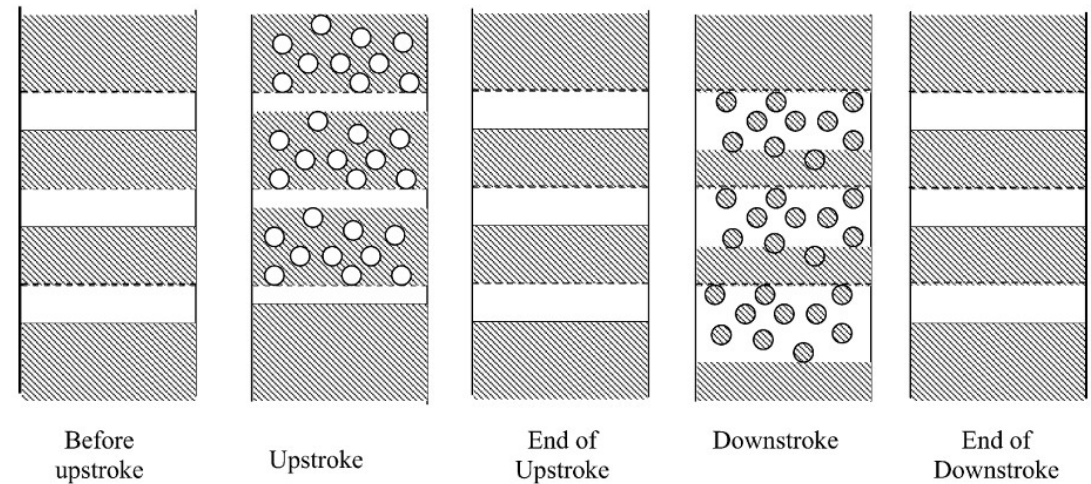


Fig: 2.2: Typical operating characteristics of pulse column

At a given throughput, flooding of a pulse column can be caused by either inadequate or excessive pulsation. Decreasing the pulse frequency causes flooding when the pulsation becomes insufficient to pass the feed streams through the column. Increasing the pulse frequency causes flooding when the pulsation

causes emulsification or the rate of counter-current flow decreases because small drop size.

Mixer settler type operation which occurs at low throughputs and low frequencies is characterized by phase separation into discrete layers during the quiescent portions of the pulse cycle as shown in Fig: 2.3. During upward pulse movement the light phase initially resting under the sieve plate is forced up through the



perforates

and rises in fairly large globules through the heavy phase layer above the plate.

Fig: 2.3: Mixer-Settler region

Similarly, during downward pulse, the heavy phase moves through the light phase zone. Mixer settler type operation is highly stable but relatively inefficient.

As higher throughputs and frequencies emulsion type operation taking place. These are characterized by small drop size and fairly uniform dispersion during the

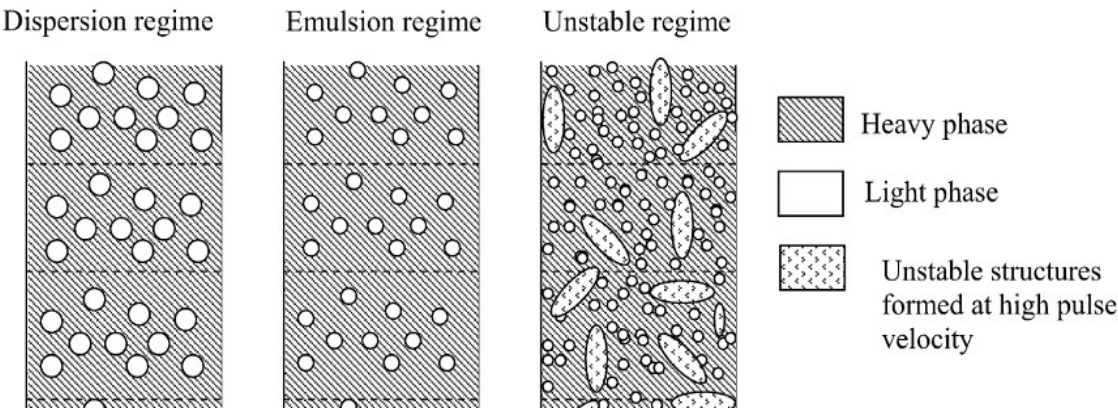


Fig: 2.4: Different flow regimes after mixer settler

course of the pulse cycle. The intimate interface contact makes this is the most efficient type of operation.

As the frequency is increased more and more, the increasing emulsification leads to two effects; (1) the holdup of the dispersed phase increases (2) the drop become smaller. These reduce the ability of the dispersed phase to flow counter to the continuous phase and finally the counter flow become impossible, the pulse frequency at which this occurs is called flooding frequency. Just below the flooded frequency is the region of unstable operation. This region is characterized by intermittent flooding over short section of the column.

Flooding capacity of a pulse column is proportional to the product of the amplitude and frequency at low frequency ranges:

These at low frequency ranges:

Pulse volume velocity is $\propto af$

Volume velocity in US gal/hr.ft² = $K af$

= 74.8 af (ref. Reactor Hand Book)

where,

a = amplitude of the pulse in inches.

f = frequency of the pulse in cpm.

K = proportionality constant 74.8

As the amplitude frequency product increases a maximum flooding volume velocity is obtained. Any further increase of af product results in the decrease of flooding volume velocity of a system. At a fixed amplitude, variations in frequency result in flooding at both a minimum and maximum frequency. For a fixed af product flow rate may be varied over a wide range with all types of phase dispersion possible as the throughput is changed. In some cases, all three types of operation are possible before flooding occurs. This generally results in emulsion type operation. For good operation throughputs of 60-80% of flooding are used.

Direct air purging is used for interphase detection in the all the pulse column. The pulsing of the columns will be by means of air, similar to the air pulsing method. The direct air purging is said to decrease the hold-up of the dispersed phase in the column; the air bubbles retained in the column prevents coalescence of the dispersed phase and thus increasing the column capacity. The air pulsing has a capacity increasing effect compared to direct air mixing.

2.2. METHODS OF ACHIEVING PULSING:

- a) Reciprocating Pulsed Column
- b) Liquid Pulsed Column.

Table 2.1: Comparison of Reciprocating Pulsed Column and Liquid Pulsed Column

SI No	Reciprocating Pulsed Column (RPC)	Liquid Pulsed Column (LPC)
1	Power Requirement is less.	Power requirement is more as the power is required to lift the liquid against gravity.
2	The minimum in HTU occurs at and near flooding, thereby improving the contactor effectiveness.	Minimum well below the flooding.
3	The axial mixing is less in RPC compared to LPC due to motion of mechanical surface throughout cross section.	The axial mixing is comparatively more due to radial variation in velocity profile in LPC.
4	Comparatively low interfacial area and low degree of turbulence.	High interfacial area and high degree of turbulence

2.3. SELECTION:

Liquid pulsed column is used widely for the following reasons:

- a) High interfacial area
- b) High degree of turbulence
- c) Low inventory required
- d) Short residence time
- e) High efficiency
- f) Low maintenance need

2.4. CONTACTOR EFFECTIVENESS

One of the basis for the comparison of the effectiveness of various types of contacting devices is the size-capacity relationship. The volume occupied by a contactor is an important variable since space requirements and fabrication costs are approximately proportional to volume. Therefore if volume is taken as the variable, then a quantitative measure of the contactor effectiveness is expressed as:

$$\text{Contactor Effectiveness} = K \frac{(\text{No. of theoretical stages})(\text{Vol. Flow rate})}{\text{Volume occupied by contactor}} = K \frac{N.F}{V}$$

Where,

F = volumetric flow rate

V = Volume of contactor

K = A dimensionless constant

Table below gives effectiveness of various contactors,

Table 2.2: Effectiveness of various contactors

Contactor Name	Contactor Effectiveness (hr ⁻¹)
Pump mix mixer-settler	54
Packed column	
Pulsed	33
Not pulsed	12
Pulsed column	65
Centrifugal contactor	> 30-40 times of mixer-settler

2.5. DESCRIPTION:

In addition to the column shell itself, pulsed columns are provided with feed distributors, phase disengagement sections, interface control instrumentation, internal fittings and some means of generating the pulse. A schematic diagram of liquid-liquid pulsed column is shown in Fig.2.1. The liquid are fed continuously to the column, flowing counter-currently, and removed continuously from opposite ends of the column. If the holes in the plates are sufficiently small, the phase to be dispersed, usually the less dense phase, will not flow through the column. If a pulsing action is applied at the base of column, the dispersed phase is forced through the perforations on the upstroke and the continuous phase flows downward on the down-stroke. If the heavy phase is to be dispersed, it is forced through the holes on the down-stroke and continuous light phase flows upward on the upstroke. The liquids are pulsed in cyclic manner, usually sinusoidally, so that a vertical pumping action is imposed on the counter current flow of the phases.

2.6. OPERATIONAL CHARACTERISTICS OF PULSE COLUMN:

Sege and Woodfield observed five distinct regimes of pulse column operation, depending on the throughput and the pulsing conditions. The different regions of operation are:

- Flooding due to insufficient pulsing
- Mixture settler regime
- Quasi emulsion regime
- Continuous dispersion regime or emulsion
- Unstable regime and Flooding

2.7. FLOODING DUE TO INSUFFICIENT PULSING:

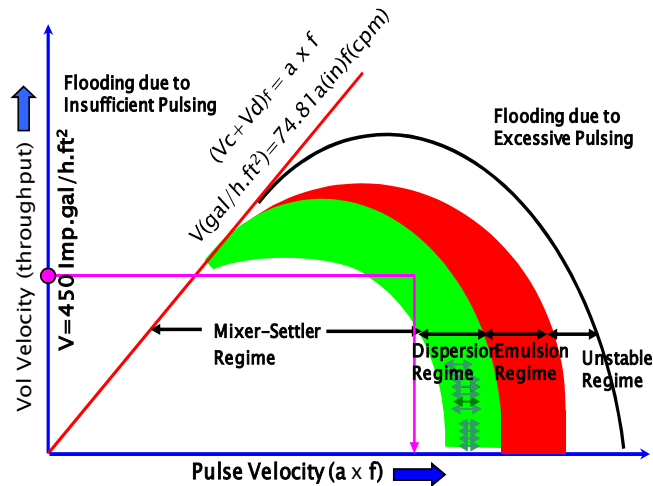
Consider a pulse column in which heavier phase (aqueous phase) is continuous phase. With no pulsing action the organic phase which enters at the bottom of the column will be prevented by interfacial tension from passing through plate perforations and will therefore forced to leave the column through the aqueous phase exit line. This condition is known as flooding caused by insufficient pulsing.

Mixer-settler regime: Mixer-settler regime occurs at low pulse velocities (A_f : 1.5 to 2 cm/s). This regime is characterized by the passes of only dispersed phase through the plate during one half of the pulse cycle and the resulting droplets tend to coalesce to form a continuous layer below the plates between successive pulses.

Continuous dispersion regime (also called quasi emulsion regime): This type of operation occurs at higher throughput and pulse velocities ($2 < A_f < 3$ cm/s). The extent of droplet coalescence between successive pulses will be much less and the column flow pattern will approximate to that of a continuous counter-current dispersion. It has been found that in continuous dispersion regime dispersed phase holdup increases:

- With increase in pulse velocity
- With increase in the flow rates of the phases
- With decrease in plate spacing, plate free area and perforation diameter
- With increase in continuous phase viscosity
- With decrease in interfacial tension and density difference between phases

PULSING PARAMETERS AND DISPERSION REGIMES



- Regimes of operation
- Pulsing parameters (a , f , duty cycle)
- Plant Capacity (throughput)

Fig. 2.5: Pulsing parameters and operating regimes of pulse column

The continuous dispersion regime is characterized by:

- Maximum rate of formation of new interfacial area
- No appreciable coalescence
- Reasonable mass transfer efficiency

Generally pulsed columns are operated in quasi emulsion regime.

2.8. UNSTABLE REGIME OF OPERATION:

Unstable region of operation occurs as the throughput or the pulsed velocity is increased to values just less than flooding.

- This is usually characterized by periodic coalescence of dispersed phase, which results in the formation of large irregular shaped globules.
- The mass transfer efficiency in the unstable region is poorer than emulsion type operation.

2.9. FLOODING REGIME:

Flooding occurs at higher pulse velocities ($Af > 3 \text{ cm/s}$). Increased pulse frequency has two principal effects:

- The holdup of the dispersed phase increases and
- Drops become smaller. As the holdup increases, the fraction of the column cross-section available for flow of the continuous phase is reduced and velocity of this phase must increase. This factor coupled with the reduced terminal settling velocities of the drops can lead to appearance of dispersed phase in the continuous phase outlet. This phenomenon is called flooding.

2.10. HYDRODYNAMICS OF A PULSED COLUMN:

The fundamental factors that affect the hydrodynamics of a pulsed column are as follows:

- The size of the droplets and their breakage & coalescence properties.
- The settling velocity of the droplets through the continuous phase: slip velocity.
- The holdup fraction of the fluid and specific contact area.
- The axial mixing intensity.

The profiles along the column and the time dependent variation of these factors during a transient should also be taken into account because these variations greatly affect the overall extraction performance. The flooding, holdup and axial mixing effects are shown in Fig. below:

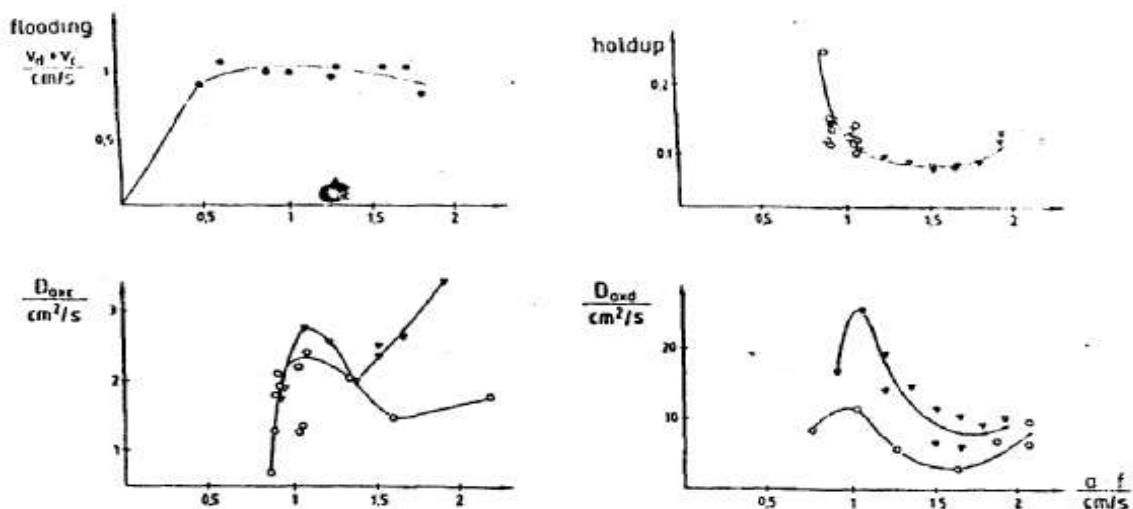


Fig.2.6: Flooding, holdup and axial mixing effects in pulsed column

2.11. HOLD-UP OF THE DISPERSED PHASE:

“Dispersed phase holdup (ε) in column is defined as the fraction of volume occupied by the dispersed phase and generally accepted to have maximum value of 0.5”.

- In a counter-current extraction column, the interfacial area available for mass transfer is dependent on the volume fraction or holdup of the dispersed phase as well as the average droplet size
- For any particular system, column geometry, and set of operating conditions, it is crucial to be able to forecast these numbers at the design stage.
- From an operational point of view, understanding the dispersed phase holdup is also crucial for inventory purposes.

2.12. EFFECT OF PULSE FREQUENCY:

The effect of pulse frequency on column holdup has been examined by Schmel and Babb for both the mixer-settler and continuous dispersion situations. The holdup of dispersed phase was found to be passing through a minimum with increasing frequency with change in the mode of operation of the column from mixer-settler to continuous-dispersion operation.

Furthermore the authors pointed out that the holdup increased with dispersed phase flow rate but it was relatively insensitive to changes in continuous phase flow rate.

2.13. CORRELATION OF HOLDUP (SEMI-EMPIRICAL RELATIONSHIPS):

In most of the published investigations, empirical or semi-empirical relationships have been proposed for correlating holdup measurements. Such procedures must be regarded as being specific to the system and the range of the variables studied. A number of correlations are available which represent holdup as a function of the product (fA) and the phase flow rates as:

$$\varepsilon = f_n(fA, U_d, U_c) \quad (1)$$

Assuming a power function for each variable:

$$\varepsilon = k (fA)^a (U_d)^b (U_c)^c \quad (2)$$

This equation being non-linear is linearized by taking logarithm and subsequently performing least square analysis for minimizing error leading to determination of constants in above Eq. (2). The correlations obtained by above procedure cannot be extrapolated beyond their experimental ranges since values of holdup at the limiting phase flow rates can become unrealistic.

2.14. THE CONCEPT OF SLIP VELOCITY:

In the theoretical treatment of counter current flow in column, the concept of the slip velocity has proved particularly helpful. The slip velocity (U_{slip}), which is the relative velocity of the dispersed phase to that of continuous phase and is given by a summation of linear velocities of the dispersed and continuous phase and is given by:

$$U_{slip} = \frac{U_d}{\varepsilon} + \frac{U_c}{(1-\varepsilon)} \quad (3)$$

The slip velocity is a function of the drop size, physical properties of the liquid-liquid system and has been found to depend upon holdup itself.

Gayler and Pratt based on the work in packed columns suggested the following approach to correlate the slip velocity:

$$U_{slip} = \frac{U_d}{\varepsilon} + \frac{U_c}{(1-\varepsilon)} = V_0(1-\varepsilon) \quad (4)$$

where V_0 is a characteristic velocity, which is mean droplet velocity relative to the continuous phase when $U_c = 0$ and $U_d \rightarrow 0$. This quantity depends upon the system properties and the pulse frequency and amplitude. The term $(1-\varepsilon)$ in Eq. (4) describes the slow down of the droplets because of smaller density difference in the two-phase system compared to the pure continuous phase. In deriving Eq. (4) it is assumed that the mean velocity of droplets relative to the continuous phase is proportional to $(\rho_m - \rho_d)$, where ρ_m is the mean density of the mixed phases in the column. Substituting for ρ_m in terms of the dispersed phase holdup and the densities [i.e. $\rho_m = (\rho_c - \rho_d)(1-\varepsilon)$], thus accounting for the term $(1-\varepsilon)$ on the right-hand side of Eq. (4).

Pratt proposed that by plotting $U_{slip}\epsilon$ vs. $\epsilon(1 - \epsilon)$ in accordance with the Eq. (4). The slope of the resulting line passing through origin gives estimate of V_0 (provided V_0 is constant over the range of holdup concerned.).

The chief criticism of the procedure is the assumption that droplet motion is represented by Stokes law. This is certainly not the case for Reynolds numbers in excess of two when more complex expressions are known to apply.

Other alternate forms of correction factors have been proposed by Misek is:

$$U_{slip} = V_k (1 - \epsilon) \exp(b\epsilon) \quad (5)$$

and by Godfrey and Slater

$$U_{slip} = V_k (1 - \epsilon)^m \quad (6)$$

Where V_k is the characteristic velocity and not necessary same as V_0 . Constant b in Misek's is accounted for the coalescence effect and in Slater's equation $U_{slip} = V_k (1 - \epsilon)^m$, the index m takes into consideration of non-idealities such as wall effects, viscosity effect in real multi-particle system.

Since characteristic velocity depends upon the system properties and the pulse frequency and amplitude, then for a given column operating in dispersion regime, the characteristic velocity defined by Eq (4) can be represented by:

$$V_0 = C_1 \exp(-C_2 f A) \quad (7)$$

Where constants C_1 and C_2 depends on the system properties. Combining Eq. (4) & (7) results following equation:

$$U_{slip} = \frac{U_d}{\epsilon} + \frac{U_c}{(1 - \epsilon)} = (1 - \epsilon) C_1 \exp(-C_2 f A) \quad (8)$$

This equation may be used to explore the way in which holdup varies with phase flow rates, frequency and amplitude.

2.15. HOLDUP AT FLOODING:

Flooding means the collapse of the two-phase counter-current flow due to increase in feed flow of one or both the phases. At the flooding point, hold up becomes maximum, and hence differentiating Eq. (3) with respect to ϵ , treating U_d and U_c as dependent variables, and putting the differentiations equal to zero, the following expressions are obtained:

$$\frac{dU_d}{d\varepsilon} = 0, \quad U_{cf} = V_0(1-2\varepsilon_f)(1-\varepsilon_f)^2 \quad (9)$$

$$\frac{dU_c}{d\varepsilon} = 0, \quad U_{df} = 2V_0\varepsilon_f^2(1-\varepsilon_f) \quad (10)$$

Solving for ε_f , the limiting value of ε at the flooding point,

$$\varepsilon_f = \frac{(L_R^2 + 8L_R)^{0.5} - 3L_R}{4(1-L_R)} \quad (11)$$

Where L_R is the flow ratio at flooding, U_{df}/U_{cf} . Thus at flooding point the holdup is a function of the flow ratio only. Eqs. (9) and (10) are used for predicting the limiting values of flow rates as other approaches zero.

- When $U_{cf} \rightarrow 0$, then from Eq. (9) $\varepsilon_f \rightarrow 0.5$ and $U_{df} = V_0/4$ [from Eq. (10)].
- Similarly, as $U_{df} \rightarrow 0$, $\varepsilon_f \rightarrow 0$ and $U_{cf} = V_0$ in the limit [from Eq. (9)].

Flooding velocity play a decisive role in calculating the column diameter. A number of correlations for these velocities are given in literature.

2.16. DROP SIZE AND INTERFACIAL AREA:

Measurement of drop size is done generally using photographic technique. In the dispersion regime the variation of droplet size with holdup is linear in those circumstances where inter-droplet coalescence alone is responsible for this effect, when gross coalescence occurs due to plate wetting and or Marangoni effects, large droplets are produced whose mean size is substantially independent of holdup. Both types of behaviour can be accommodated by an equation of the form:

$$d_{vs} = d_{vs}^0 + k\varepsilon \quad (12)$$

The constant k is the characteristic of the system and is a measure of the rate of change of droplet size with holdup. When gross coalesce is present, the constant

k becomes very small and d_{vs} approaches the value d_{vs}^0 . Where d_{vs}^0 is termed as the droplet characteristic size and may be regarded as the size of droplet having a relative velocity V_0 . For a specific column and system, the characteristic droplet size is a function of the system properties and the pulsing parameters so that d_{vs}^0 is represented by

$$d_{vs}^0 = C_3(fA)^{-C_4} \quad (13)$$

Combining Eq. (12) and (13) results in the expression

$$d_{vs} = C_3(fA)^{-C_4} + k\varepsilon \quad (14)$$

Eq.(14) enables the mean droplet size to be computed for any given set of operating condition, the appropriate value of ε being calculated from the holdup relation Eq. (8). The specific interfacial area is given by:

$$a = \frac{6\varepsilon}{d_{vs}} \quad (15)$$

2.17. MASS TRANSFER IN A PULSED COLUMN:

From the usual definition of HTU:

$$HTU_{oc} = \frac{U_c}{K_{oc}a} = \frac{d_{vs}U_c}{K_{oc}\varepsilon}$$

$$\text{i.e. } HTU_{oc} \propto (U_c d_{vs})/\varepsilon$$

The maximum rate of mass transfer is obtained by achieving the optimum combination of the maximum interfacial area, the maximum rate of formation of new surface area and maximum degree of turbulence in both phases. If the phenomena that accompany increasing pulse frequencies are examined, it is found that the interfacial area is a maximum in the region of unstable operation or at emulsion flooding but that the maximum rate of new surface generation occurs with quasi-emulsion operation. Very little surface is generated in the region of unstable operation. Turbulence in the continuous phase is probably satisfactory at all frequencies above the middle of the mixer-settler region. In the discontinuous phase, however the turbulence decreases at high frequencies. Small droplets are known to have little internal circulation, they transfer mass to the interior chiefly by molecular diffusion, whereas large drops transport mass freely by internal circulation.

In addition to the efficiency features, two sources of inefficiency are encountered: channelling and back mixing. Channelling can be counteracted by ensuring a good fit between sieve plates and the column wall and by providing for redispersion in large columns by means of louver plates.

Back mixing in a pulsed column is unavoidable by the very nature of things and

undoubtedly contributes much to unsatisfactory prediction of extraction performance. The theory of back mixing is now receiving considerable attentions, but experimental studies of back mixing in pulsed columns are limited. Back mixing is known to cause curvature of the operating line, so that more stages or transfer units are required for a given separation than would be expected from the terminal conditions. The effect of back mixing is greatest for substances having high extraction factor for extraction and low extraction factor for stripping.

Burger and Swift found that the rate of back mixing depended largely on the continuous phase flow rate and to a lesser extent on pulsed amplitude, plate spacing or dispersed phase flow rate. Mar and Babb found that back-mixing increased with an increase in plate spacing, pulse frequency and perforation diameter and that it was greater when the continuous phase did not wet the plates.[10]

2.18. DESIGN CONSIDERATIONS FOR PULSED COLUMN:

2.18.1. DESIGN AND OPERATION OF A SOLID EXTRACTION COLUMN IS DECIDED BY DROP SIZE.

Drop size affects on:

- ✓ Disperse phase hold up
- ✓ Residence time of the dispersed phase
- ✓ Allowable throughput/capacity of the column
- ✓ Mass transfer
- ✓ Phase separation
 - Determining the column diameter and column height is necessary for extraction column sizing.
 - The column's diameter needs to be large enough to allow the two phases to flow against each other without flooding.
 - To obtain the number of equilibrium phases corresponding to the desired degree of separation, the column height must be sufficient.
 - A precise assessment of the column diameter for a liquid-liquid contactor is difficult and unpredictable due to the numerous variables.
 - Individual phase flow rates, the difference in densities between the two phases, interfacial tension, the direction of mass transfer, the viscosity and

density of the continuous, the speed of rotation or reciprocation, and internal geometry are a few examples of variables.

- The easiest way to evaluate column diameter is by scaling up test runs in pilot plant or laboratory test units with a diameter of roughly 1 in. Then, it may be expected that bigger commercial units will maintain the sum of the recorded surface velocities of the two liquid phases in the test unit.

2.18.2. CARTRIDGE TYPES AND GEOMETRY:

The assembly of plates in a liquid pulsed column is called the plate cartridge. There are three main types of cartridge:

- Dual-faced plates
- Stainless steel nozzle plates
- Mixed-plate cartridge

2.18.2.1. DUAL-FACED PLATES:

They are stainless steel sieve plates coated with plastic on the side facing the organic-phase inlet to the column. These plates have been successfully used to give high capacity and efficiency with either phase continuous. A disadvantage of this plate is the question of durability of plastics in radioactive fields.

2.18.2.2. STAINLESS STEEL NOZZLE PLATES:

These are a special class of sieve plates, are characterized by the indentation of holes to form many tiny jetting nozzles. These are usually oriented towards the dispersed-phase exit of the column. A well-designed nozzle plate cartridges have capacities about 50% higher and HTU up to 30% lower than equivalent geometry flat sieve plates.

2.18.2.3. MIXED-PLATE CARTRIDGE:

These are basically stainless steel sieve plates or nozzle plate cartridges with plastic plates introduced at frequent intervals, have been used in both aqueous and organic continuous in columns. The usual objection to the use of plastics in cartridges is their inability to withstand high radiation doses.

Many studies of plate design and its effect on column performance has led to the use of “standard geometry”; plates with 3mm diameter holes on a triangular array to give 23% free area are spaced 50mm apart in the column (plate thickness of up to 2mm are usual). The range of plate cartridge geometry used is shown in **Table 2.3**

Table 2.3: Range of plate cartridge geometry

Plate diameter	10 – 2500 mm
Orifice diameter	0.83 – 4.78 mm
Free area	10 – 60%
Plate spacing	12.5 – 100 mm
Nozzle length	1 – 1.5 mm

2.18.3. CHOICE OF CONTINUOUS AND DISPERSED PHASE:

It is nearly usually up to the equipment designer to decide which of the two liquids will become the scattered phase. For a given droplet size, it is typically preferable to spread the liquid that flows at a higher volumetric rate because this will result in a larger interfacial area. However, there are other factors that can occasionally take precedence when choosing this option:

- If the ratio of volumetric flow rates is very greatly different from unity it will be preferable to disperse the minority liquid in order to minimize back mixing.
- The dispersed phase normally has the smaller holdup in column. For reasons of inventory cost and fire hazard, it is more desirable to disperse the more costly or more flammable liquid.
- Mass transfer rate is greatly influenced by the direction of mass transfer. Mass transfer rate is higher in the case of C → D transfer whereas in D → C mass transfer case coalescence rate is faster.
- The liquid, which wets the material of construction of column should made as continuous phase in order to increase throughput and avoid channelling.

2.18.4. COLUMN CROSS-SECTIONAL AREA:

Once numerical value of V_0 is established, the column cross/sectional area required for a given throughput may be calculated from either Eq. (9) or (10), the holdup at flooding being obtained from Eq. (11). Thus for example if it is desired to calculate the size of column to accommodate flow rates of L_c and L_d m^3s^{-1} respectively, the column cross-sectional area A_f m^2 which will just permit the passes of this quantity of fluid is given by:

$$A_f = L_d/U_{df} = L_d/2V_0\varepsilon_f^2(1-\varepsilon_f) \quad (18)$$

Where ε_f is calculated from Eq. (11) by setting the flow ratio LR to L_d/L_c . The actual column cross sectional area employed is then taken to be equal to $2A_f$ so that column operates at 50% of flooding.

2.18.5. COLUMN LENGTH:

Mass transfer calculations are needed to determine the column height, which requires knowledge of the number of theoretical stages or transfer units from equilibrium distribution data in terms of stage efficiency, mass transfer coefficients and finally the correction necessary for axial mixing. Unfortunately, all these factors are influenced by the size of droplets in the system and their associated hydrodynamic behaviour.

2.18.6. PULSE GENERATION:

Pulse can be generated by two different ways. These are:

- Mechanical pulse generation
- Pneumatic pulse generation

2.18.6.1. MECHANICAL PULSE GENERATION:

Here mechanical pulser is used for generation of pulse. Mechanical pulse generators are of three types: piston, diaphragm and bellow. The pulser is hydraulically attached to the least radioactive stream entering or leaving the base of the column; leaked fluid can be collected and returned to the process or sent to the solvent recovery system or to the waste system.

2.18.6.2. PNEUMATIC PULSE GENERATION:

Here, pulse is generated by periodic application of air pressure. Radioactive contamination of pulsed air is minimized by pulsing the stream at the bottom of the column that is least radioactive; this is the entering organic stream. The pulse leg is attached as close to the base of the column as feasible.

3. AIM AND SCOPE

The methodical investigation of pulse column operational factors, such as air reservoir pressure, pulse amplitude, pulse frequency, light liquid flow rate, and heavy liquid flow rate, which affect flooding..This experiment help us to know basic understanding of flooding.

WIP, BARCF,Kalpakkam is going to use Pulsed column for recovery of trace amount of uranium there in the HLW. This experiment was a part of the pre-commissioning study. Now with the help of these pulsing parameter, we can operate the pulsed column in actual case for HLW. Since, in actual case only the fluid will be changed, so only the hydrodynamic parameters will be changed and we can comfortably use the pulsing parameters in real situations.If it is within the allowable range i.e. 1%-2%, then our experimental results are perfect.

4. EXPERIMENTAL DESCRIPTION

4.1. DESCRIPTION OF EQUIPMENT AND EXPERIMENTAL SETUP

The as built drawing of the pulse columns used in the study is presented in Fig:4.1..The pulse column had a 50 mm diameter, 40 plate count, and 50 mm plate spacing. The column was made of plates with a 23% free area that were sandwiched between sections of flawless glass. The contacting section of the column was 2000 mm high. End section of the columns were made of 100 mm diameter glass section. The nozzle and sieve plate were made of SS304L and their details are presented in Fig:4.2. The pulsing leg of the column was made from 25 mm diameter glass tube. The contacting section of the columns were provided with sampling ports at different heights for withdrawal of samples.

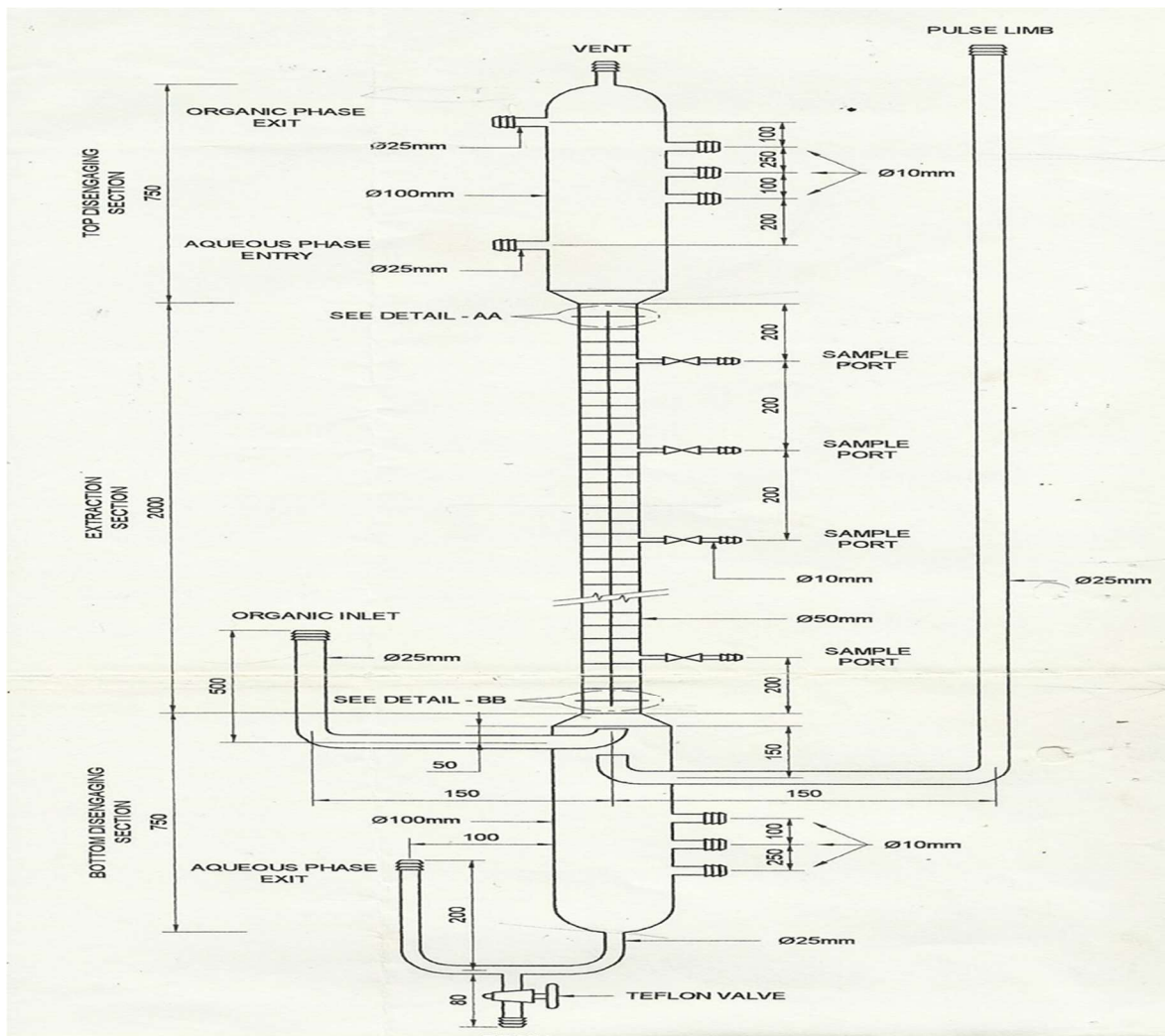
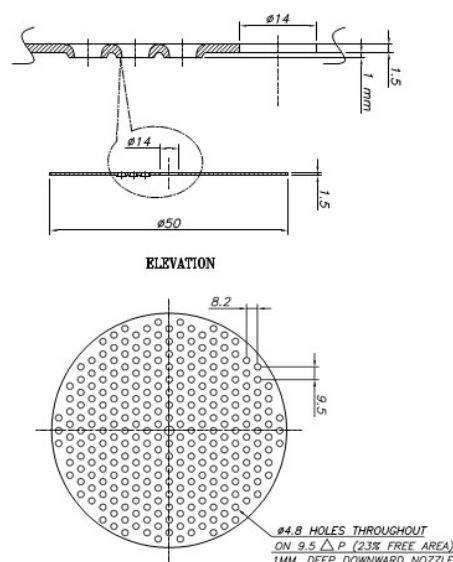


Fig: 4.1: Asbuilt of Experimental Pulse Column

Table 4.1: Pulse Column Details

Details	3A
Type of plate	Nozzle
No. of plates	40
Gap between plates	50 mm
Plate thickness	1.5 mm
Opening Dia.	4.8 mm
Holes Pitch	9.5 mm
Height of Extraction section	2000 mm
Dia. of Extraction section	50 mm
Height of top disengagement Section	750 mm
Dia. of top disengagement Section	100 mm
Height of bottom disengagement Section	750 mm
Dia. of bottom disengagement Section	100 mm
No. of sampling port	6

**Fig. 4.2: Nozzle Plate Detail**

The pulsing of the column contents was carried out using compressed air and consisted of air reservoir, PRV and 3-way solenoid valve. The schematic of the pulsing system is shown in Error! Reference source not found.. The pulse air was introduced through bottom of the column below the organic phase feed point. It takes amplitude and frequency to determine a pulse's characteristics.. "The pulse

amplitude is defined as the total linear displacement of the fluid contents in the column cross- section, from one extreme position to other. The product of the pulse amplitude and the column cross-sectional area is called the pulse volume, while the product of amplitude and frequency is called pulse intensity”.

The pulse amplitude was measured visually by observing the movement of the fluid meniscus in the pulse leg with the help of graduated scale. The measured pulse amplitude was cross checked by measuring the total linear displacement of the fluid directly in the column.

Using a two stage air lift system, the organic and aqueous phases were fed from the bottom and top, respectively. The schematic of the experimental setup is shown in Fig: 4.3 and the actual set up is shown in Fig: 4.4

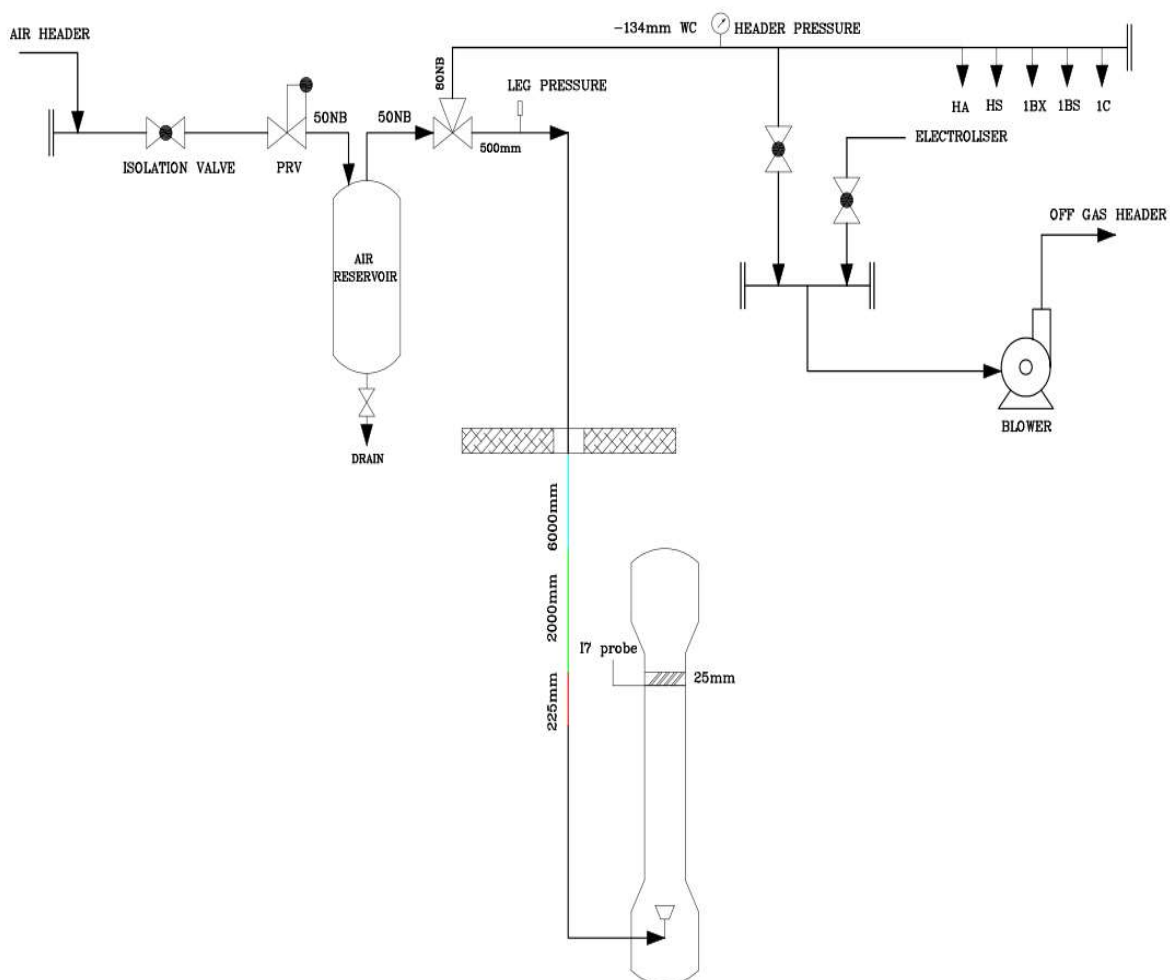


Fig: 4.3: Schematic of pulsing set up



Fig: 4.4: Experimental pulse column and bottom disengagement section

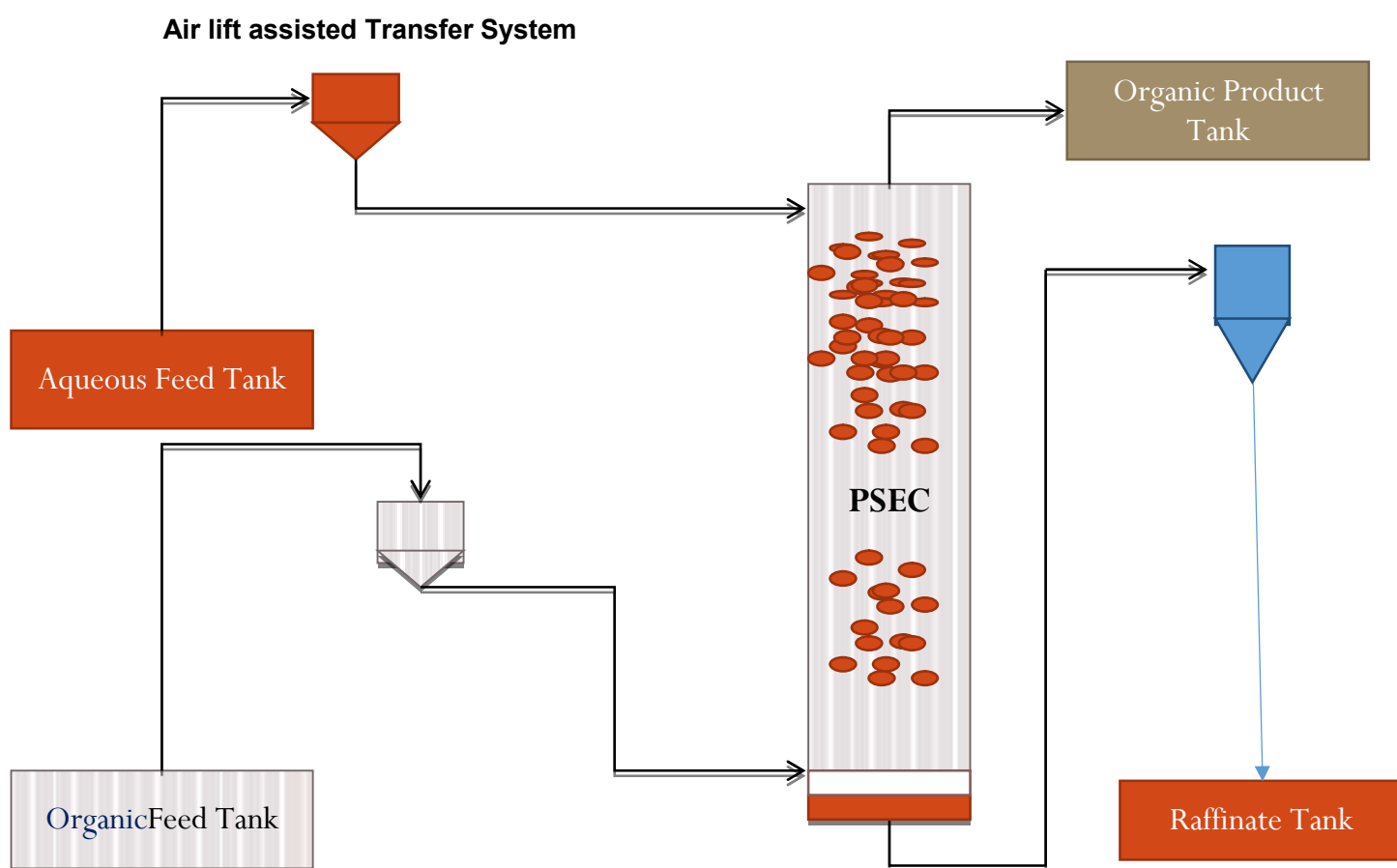


Fig: 4.5: Schematic diagram of experimental setup

4.2. ROSS VALVE:

Ross valve is a three way valve which is used for the generation of pulse with the help of instrumentation air. The Ross valve in field is connected to a timer in the control room. The valve causes direct pass flow and side pass flow respectively as per the command given to it from the control room with the help of the timer.

Position 1: 1-3 connected means Pulse is ON ie, air goes to the pulsing leg. When the pulsing action is required, the pressure switch actuated the solenoid valve, which opens inlet and outlet and air flows through pulse leg.

Position 2: 2-3 connected means pulse is OFF, ie air is venting out to the VOG (Vessel Off Gas) System. In order to get the liquid column exactly to its original position, the line pressure should become zero. For this reason, the exit port of the solenoid valve is connected to off gas line.

position 1
direct pass flow

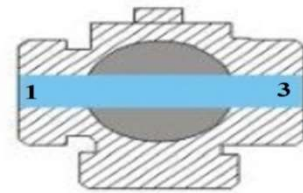


Fig: 4.6: Position 1: Direct Pass flow

position 2
side pass flow

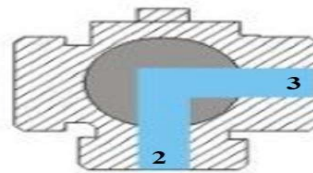


Fig: 4.7: Position 2: Side Pass flow



Fig: 4.8: Three-Way solenoid valve (Ross valve)

4.3. AIR LIFT PUMP:

Air is admitted to the transfer line. This air mixes with the liquid in the transfer line and forms a mixture of lower density. Due to this the comparatively high-density liquid in the column pushes the liquid and air mixture upward. This liquid and air mixture enters the ALP (Air Leak Path) tank where the liquid and air are separated. The liquid is collected in the collection tank while the air leaves through off gas line. Air flow rate should be optimum, because if the flow rate is high some amount of liquid will be carried over by the air (entrainment) on the other hand, if the flow is less, the density of the air and liquid mixture will not be sufficient to lift through the transfer line.

Two critical factor of air lift pumping are:

- The submergence of the air line: Submergence always mean the depth of air line below the pumping level and
- Size of discharge line

4.3.1. AFFECTING VARIABLES:

Expected to affect a lift performance are:

- Pipe diameter
- Lift pipe length
- Percentage submergence
- Liquid density
- Liquid temperature
- Foot piece and nozzle design

The lift capacity increases as the pipe diameter is increased but not in direct ratio, there is a definite minimum lift pipe diameter for each set of operating condition.

The percentage submergence is probably the most significant variable. The lift capacity always increases with increased submergence.

4.4. METHODS, TECHNIQUES AND SYSTEM IMPLEMENTATION:

4.4.1. SENSING METHODS:

For the measurements of level, density and pressure air purge method is used. This is a non-contact method and radioactive process fluids do not come in contact with the sensor.

Electronic Smart transmitters with suitable range are used for different measurements. Transmitters are selected such that the measurement range falls nearly in the middle/two-third of the transmitter range. Different ranges are selected to obtain better resolution. Smart devices allow remote and in-situ calibration.

Differential pressure transmitters are centrally located in the transmitter rack area. They are located at higher elevation than the process equipments to avoid any back flow of process liquids and reaching the transmitters. Two probes are used for level and density measurements in closed in-cell equipments. Single probe is used for pressure measurement. A constant flow rate of around 20 NLPH of air at 1.4kgf/cm² is maintained for purging. This flow rate is monitored and controlled by means of purge rota meter with an integral needle valve and pressure regulator. The backpressure is taken to the differential pressure transmitter mounted on the transmitter rack by means of 6mm OD, 1mm thick SS304L tubes.

For the level and density measurements, the transmitters are suitably calibrated to the range of measurements. All these parameters are measured in terms of pressure using the relation:

$$P = h \rho g$$

P= Back Pressure

h= Liquid level/distance between the probes

ρ = Density

g=the acceleration due to gravity

For level measurements, the density is assumed constant and hence,

$$P \propto h$$

Level transmitters are calibrated from range $h=0$ to $h=h_{max}$. Whenever it is necessary to have a correction of level variation due to change in density, it is achieved by suitable configuration in the PLC/ Video Graphic Recorder. This is done dynamically using math function in PLC and recorders, which accepts both the level and density as parameters. Only the corrected level is displayed. Typical range of level measurement is between 500mm and 2000mm. Level is displayed and recorded in percentage values.

In the case of density transmitters, the HP and LP probes are designed such that both the probes are immersed in process fluid where density is assumed to be

uniform. For density- measurement, h is kept constant typically 300mm. If d_1 and d_2 are minimum and maximum densities, the transmitter is calibrated from

$$P_1 = h d_1 \text{ to } P_2 = h d_2$$

Typical range of density is from 0.7 to 1.4 g/cc. Density of process fluids are displayed and recorded in g/cc units.

For pressure measurement in closed vessels, the range is -300 to + 300mm WC [14]

4.4.2. SIGNAL TRANSMISSION:

The transmitter output signal, which is standard 4-20 mA DC, is given to PLC as analog input. Liquid level signals are duplicated and fed to VGRs in control room. Two-wire transmission is used for signals to be transmitted.

Signal Distributor/Isolator (SDI) units are used in analogue loops to provide 24 V DC power supply for transmitters, signal isolation and for distribution of process signals to different receiving instruments. [5]

4.5. WATER RUN FOR ESTABLISHING RESERVOIR PRESSURE AND PULSE AMPLITUDE

4.5.1. PROCEDURE

1. The experimental setup area is allowed to receive DM water from the DM water plant.
2. The 1AF water storage tank, which is close to the pulsing column
3. The ALPs are activated to fill the pulsing column with water from the bottom when the water storage tank level reaches 80%.
4. The ALPs are halted when the water column level reaches almost 2000–2200 mm.
5. Permit the level to steady for a while.
6. The Ross valve is disinfected and made operational.
7. We strive to identify the desired pressure at which the ideal circumstances are attained in order to prevent reservoir air from entering the column when it is pulsing.
8. Keeping the duty cycle constant at 40%, which is the optimum value for a pulsed column, at different frequencies, we tried to find out the optimum pressure required.

9. The pressure is that fixed in that optimum value and at different frequencies and duty cycles we carried out the experiments to note down the changes in amplitude.
10. The pulse amplitude was measured visually by observing the movement of the fluid meniscus in the pulse leg with the help of graduated scale.
11. The measured pulse amplitude was cross checked by measuring the total linear
12. Displacement of the fluid directly in the column.
13. We then tried to find out the relationships amongst the parameters like, amplitude, frequency, and air reservoir pressure.

After the initial determination of reservoir pressure and pulse amplitude during water trials, the pulsing study of the column using two phase system was carried out. In these studies, 20% TBP in n-Dodecane was used as organic phase and water was used as aqueous phase. The purpose of the study was to establish the flow regimen curve for TBP in n-Dodecane and water system.

4.5.2. PROCEDURE FOR FINDING FLOW REGIME AND FLOODING

1. Before performing column runs, the aqueous and organic phases were repeatedly pumped through the column to reach mutual saturation.
2. Aqueous effluent stream passed from the bottom of the column using an airlift and was collected in a 100-liter capacity tank. Aqueous feed stream entered the top of the column used as demineralized water.
3. The organic effluent stream exited the column at the top and was collected in a 100-liter tank. The organic feed stream entered the column at the bottom and was made up of 80% tributyl phosphate and 20% n-dodecane.
4. The pulse column was normally activated at a certain pulse frequency once flow rates were adjusted to the proper values.
5. Using a purging approach based on density difference, the main interface was kept at the bottom of the column.
6. The continuous phase flow rate was adjusted to a desired condition and the dispersed phase flow rate was gradually increased until it reached the required value at constant amplitude and the duty cycle was fixed at 40%.

7. The column was assumed to be at steady state in the usual operating zone once the flow rates, the Interface level, and the differential pressure were constant for at least 30 minutes.
8. The frequency was then varied in order to obtain different flow regimes at a given aqueous and organic flowrate.
9. The gradual increment in the frequency of pulsation was carried out till flooding was achieved.
10. While gradually increasing the frequency , visually inspecting the column to determine which flow regime is operating.
11. The fluctuation in column differential pressure, column's interphase, and a change in column density are all evident signals for flooding.
12. At the bottom of the column, the interface rises uncontrollably, and a new interphase forms at the top. These are reliable signs that flooding has replaced the previous state of functioning.
13. Also seen was the accumulation of an organic phase layer in the bottom plate's terminal portion.
14. The flow rates at the start of flooding were calculated using the effluent tank levels.
15. The column was then stopped in order to measure the pulse volume.

5. RESULTS AND DISCUSSION

The columns were operated with Demineralised Water for establishing the air reservoir pressure, frequency and duty cycle for achieving desired pulse amplitude. Different set of data recorded to achieve the desired amplitude are presented in After the initial determination of reservoir pressure and pulse amplitude during water trials, the pulsing study of the column using two phase system was carried out. In these studies, 20% TBP in n-Dodecane was used as organic phase and water was used as aqueous phase. The purpose of the study was to establish the flow regimen curve for TBP in n-Dodecane and water system.

Number of flooding runs were made at flowrates of (Aqueous +Organic) 20-83 lph in which pulse frequencies from 0 to 200 cpm were studied and the data is presented in **Table 5.4**.

5.1. RELATIONSHIP BETWEEN AMPLITUDE AND AIR RESERVOIR PRESSURE GRAPHICAL ANALYSIS

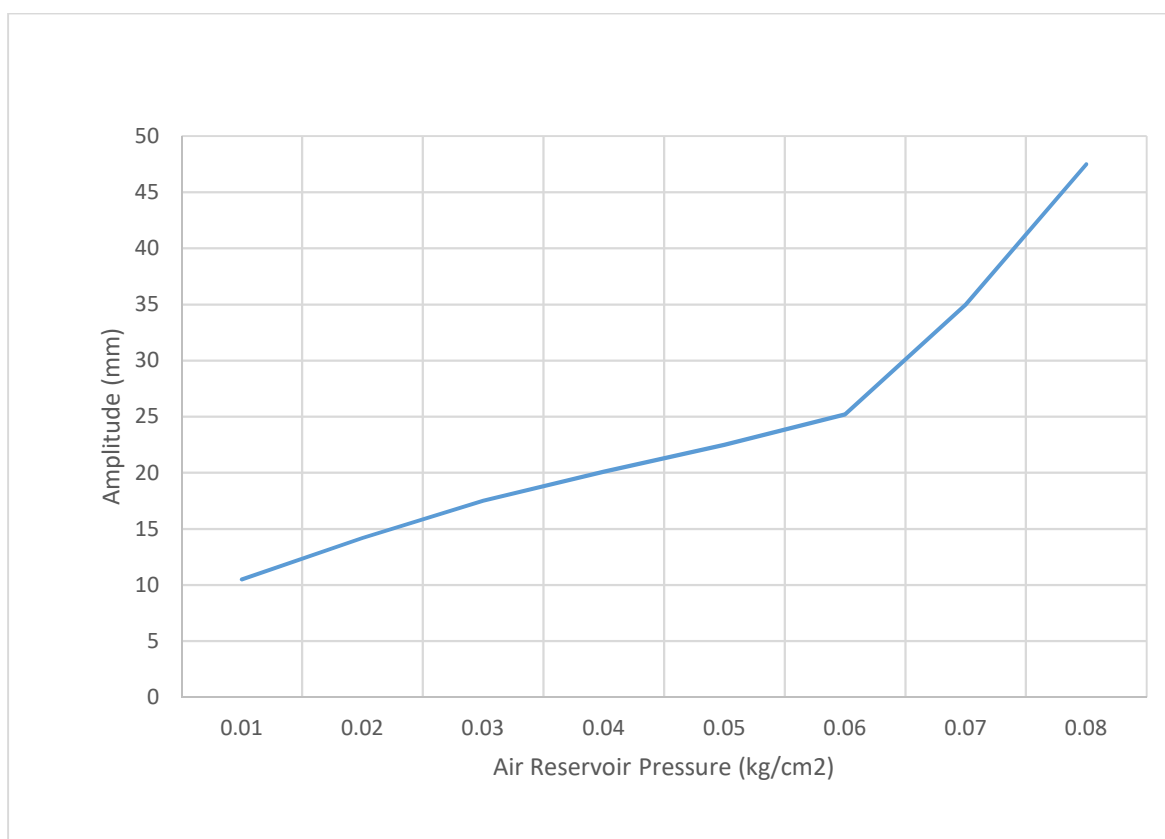


Fig. 5.1: Amplitude vs Air Reservoir Pressure

Table 5.1: Data recorded to optimize air reservoir pressure

Duty Cycle (%)	Frequency (cpm)	Air reservoir Pressure (Kgf/cm²)	Mean Amplitude(mm)	Remarks
40	60	0.08	47.5	Too high
40	60	0.07	35	Too high
40	60	0.06	25.19	Required
40	60	0.05	22.5	Low
40	60	0.04	20.1	Low
40	60	0.03	17.5	Low
40	60	0.02	14.2	Low
40	60	0.01	10.5	Low
40	70	0.06	22.1	Low
40	70	0.04	28.1	Too high
40	70	0.02	15.1	Too low
40	60	0.06	25.18	Required
40	55	0.06	30	Too high
40	55	0.02	20	Low
40	65	0.06	22	Low
40	65	0.04	20	Low
40	65	0.02	20.12	Low

5.2. DETERMINATION OF MEAN AMPLITUDE**Table 5.2: Determination of mean amplitude**

Duty Cycle (%)	Frequency (cpm)	Air reservoir Pressure (Kgf /cm²)	Amplitude (mm)
40	60	0.06	25.32
40	60	0.06	25.45
40	60	0.06	24.91
40	60	0.06	25
40	60	0.06	25.75
40	60	0.06	25.2
40	60	0.06	24.81
40	60	0.06	25

Average Amplitude: 25.18mm≈25mm

5.3. RELATIONSHIP BETWEEN AMPLITUDE AND FREQUENCYGRAPHICAL ANALYSIS

Table 5.3: Variation of amplitude with Frequency and duty cycle at constant Reservoir pressure (0.06 kgf/cm²)

Duty Cycle	Frequency	Mean Amplitude(mm)	Remark
35	50	40.5	Too High
	60	32.5	Too High
	65	13	Too Low
	70	11.25	Too Low
40	50	29.95	High
	60	25.18	Required
	65	17.85	Low
	70	14.3	Too Low
45	50	30	Too High
	60	15.5	Too Low
	65	14.2	Too Low
	70	13.25	Too Low

At Air Reservoir Pressure: 0.06 kg/cm²

Duty Cycle: 35%

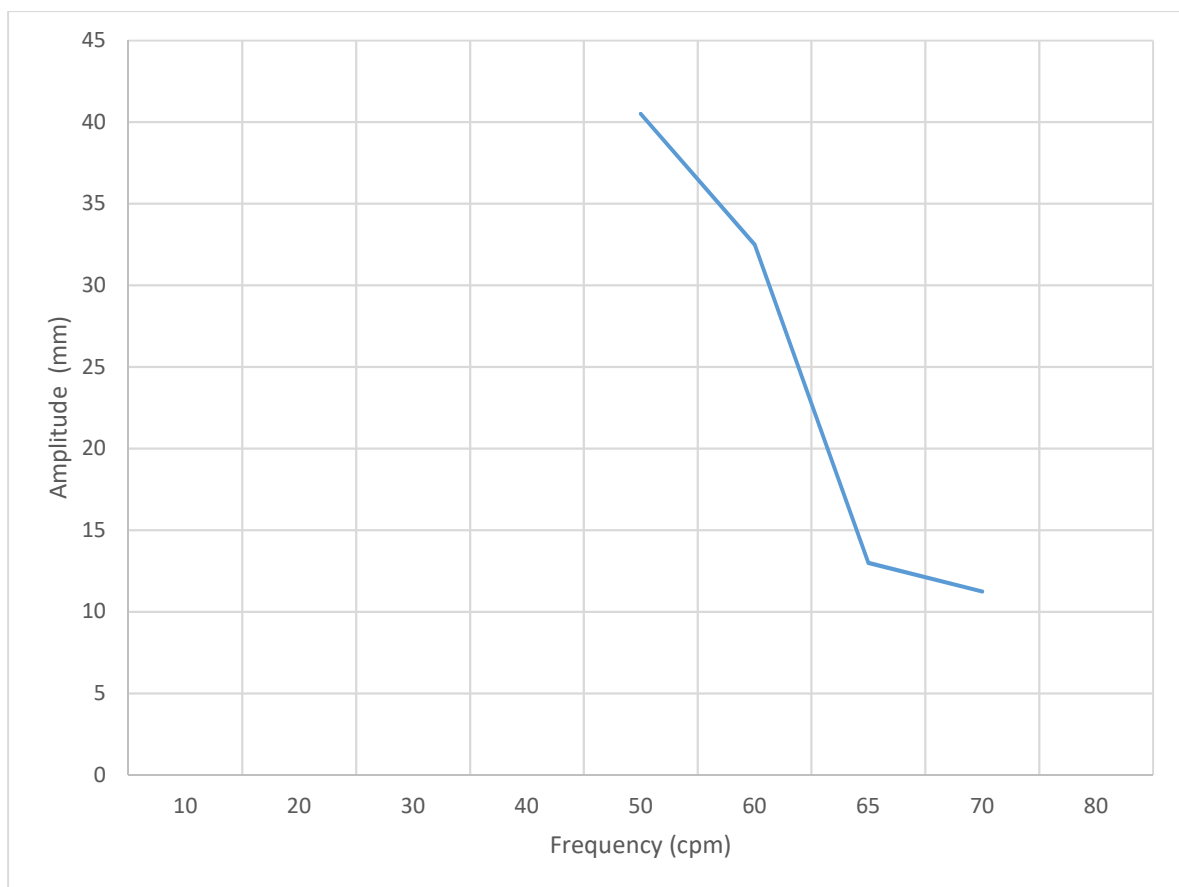


Fig 5.2: Amplitude vs Frequency at Duty Cycle 35%

At Air Reservoir Pressure: 0.06 kg/cm²
Duty Cycle: 40%

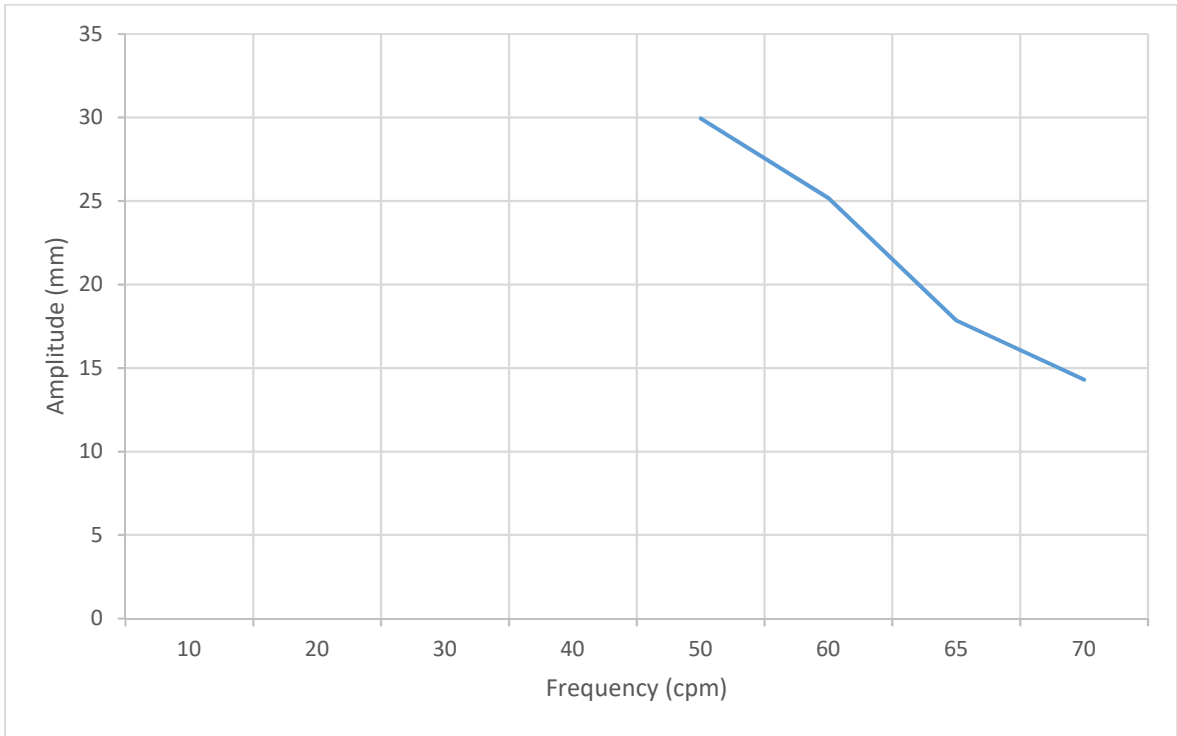


Fig: 5.3: Amplitude vs Frequency at Duty Cycle 40%

At Air Reservoir Pressure: 0.06 kg/cm²
Duty Cycle: 45%

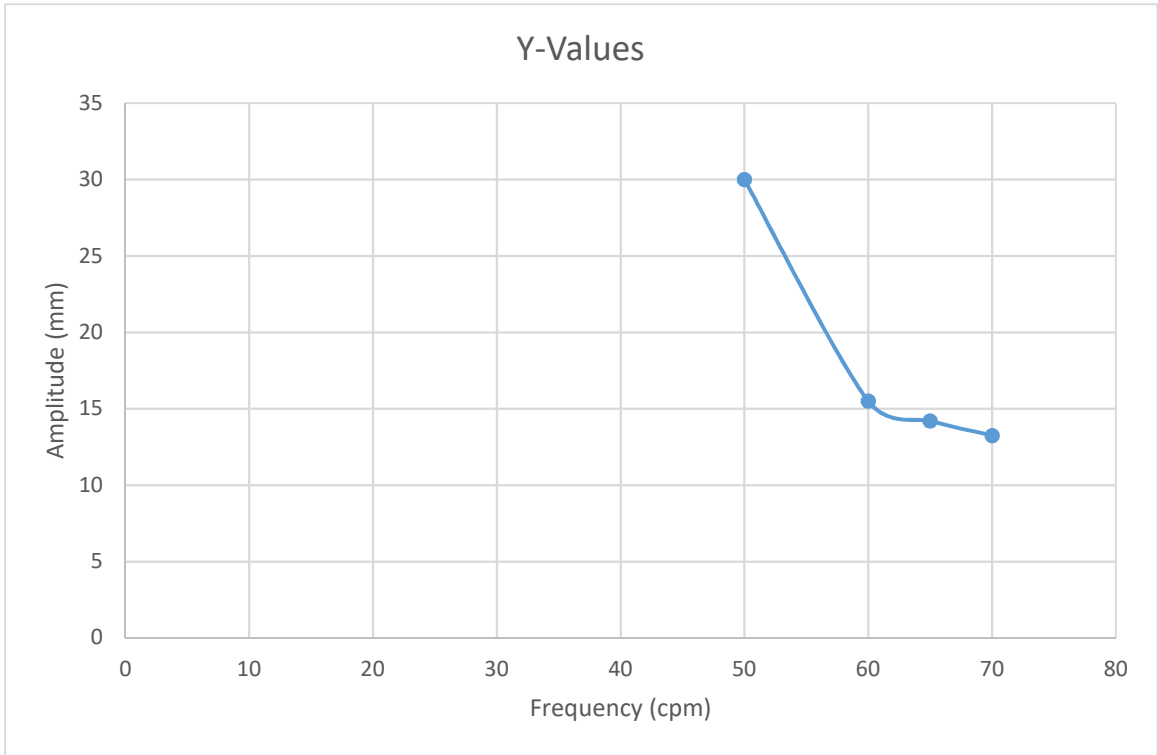


Fig: 5.4: Amplitude vs Frequency at Duty Cycle 45%

5.4. FLOODING CHARECTERISTICS OF PULSED EXTRACTION COLUMN

Table 5.4: Flow regime obtained at different frequencies and duty cycle of 40%

Sr. No	Cumulative flowrate	Frequency	Observed Flow regime	Velocity at Flooding
	LPH	cpm		mm/s
1	20	10	Flooding due to insufficient pulsing	58.33
		20 to 70	Mixer Settler	
		80 to 100	Dispersion	
		110 to 130	Emulsion	
		140	Flooding	
2	30	16	Flooding due to insufficient pulsing	54.17
		20 to 70	Mixer Settler	
		80 to 100	Dispersion	
		110 to 120	Emulsion	
		130	Flooding	
3	35	18	Flooding due to insufficient pulsing	52.08
		20 to 60	Mixer Settler	
		70 to 90	Dispersion	
		100 to 120	Emulsion	
		125	Flooding	
4	40	20	Flooding due to insufficient pulsing	50.00
		30 to 50	Mixer Settler	
		60 to 80	Dispersion	
		90 to 110	Emulsion	
		120	Flooding	
5	45	22	Flooding due to insufficient pulsing	45.83

Sr. No	Cumulative flowrate	Frequency	Observed Flow regime	Velocity at Flooding
	LPH	cpm		mm/s
		30 to 40	Mixer Settler	
		50 to 70	Dispersion	
		80 to 100	Emulsion	
		110	Flooding	
6	50	25	Flooding due to insufficient pulsing	41.67
		30	Mixer Settler	
		40 to 60	Dispersion	
		70 to 90	Emulsion	
		100	Flooding	
7	60	30	Flooding due to insufficient pulsing	37.50
		40 to 50	Dispersion	
		60 to 80	Emulsion	
		90	Flooding	
8	70	35	Flooding due to insufficient pulsing	33.33
		40 to 50	Dispersion	
		60 to 70	Emulsion	
		80	Flooding	
9	80	40	Flooding due to insufficient pulsing	25.00
		50	Emulsion	
		60	Flooding due to excessive pulsing	
10	83	50	Flooding due to insufficient pulsing	20.83
		50	Flooding due to excessive pulsing	

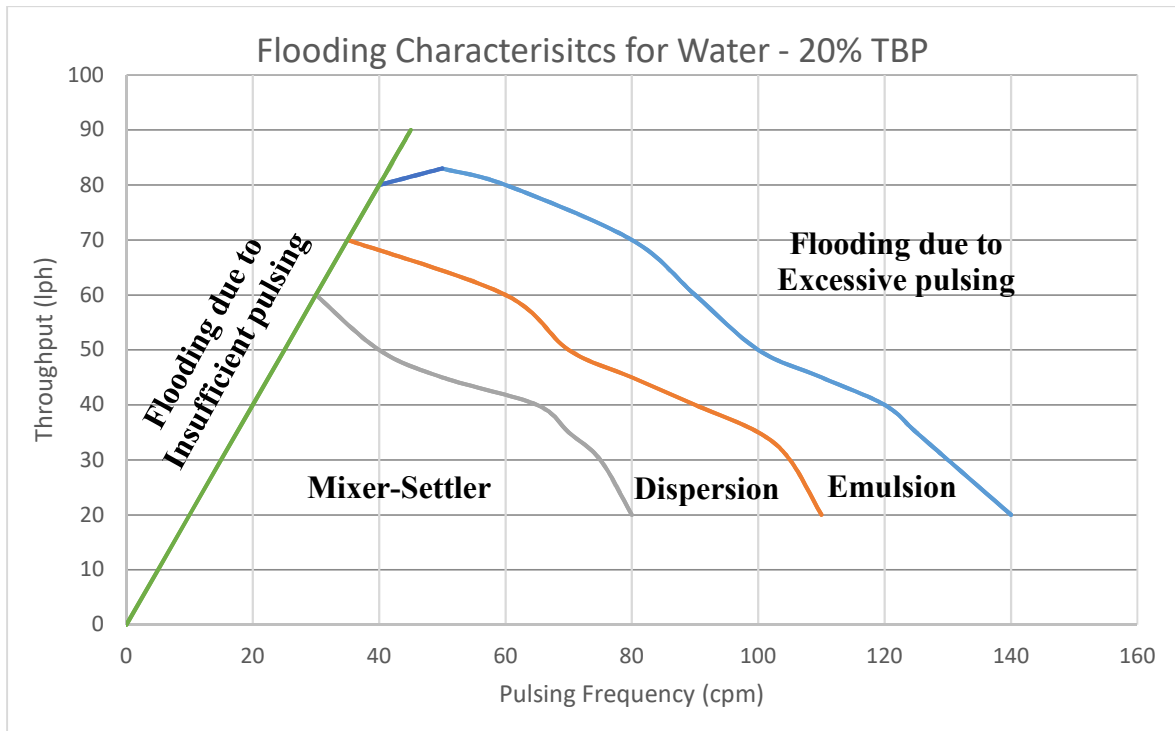


Fig: 5.5: Flooding Characteristics for Water-20% TBP

From the data recorded and the graphs plotted, it is found that we are getting an amplitude of 25 ± 0.3 mm which is half of the plate spacing, (in our case, plate spacing is 50mm) at the following set of parameters:

Duty cycle: 40%

Frequency: 60 cpm

Air reservoir pressure : 0.06 kg/cm^2

Aqueous and organic flow rate : 70 lph

Therefore, it can be said that the pulse column can be operated at the above parametric values to have a reasonable turbulence at which mass transfer and hence the extraction efficiency will reach the desirable point.

6. SUMMARY AND CONCLUSIONS

In this experiment found out the operating parameters required for specific pulse column operations with demineralised water. To determine the flooding point for this specific column, we experimented with various flow regimes in a pulsed sieve extraction column.

The following is a list of the experiment's findings:

Functional relationship: Here we tried to find out the functional relationship between amplitude and frequency, amplitude and reservoir pressure and we found out that

As frequency increases, amplitude decreases.

As pressure increases, amplitude increases.

In course of experiment it was found that slight change in pressure did not have much impact on amplitude, whereas the changes in frequency causes a change in amplitude prominently. Thus we can conclude that amplitude is a strong function of frequency and a moderate function of air reservoir pressure.

Duty cycle is kept at 40%, which is an optimum value in the context that if it is less than 40% ($D < 40\%$) the air pulse entered into the column through the pulsed leg will be so small that it will not create sufficient pulsing necessary to create turbulence. Moreover in actual case, the light phase will then come out through the heavy phase outlet (flooding due to insufficient pulsing). On the other hand, if duty cycle is greater than 40 % ($D > 40\%$) the pulse will be too high to produce the necessary amplitude and flooding due to high velocity will take place.

In this experiment found that flow regimes in pulse column are changing with frequencies and aqueous-organic flow rate.

In the course of experiment found that flooding is happening due to insufficient pulsing and excessive pulsing.

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