Experimental and Modeling Studies for Online Measurement of Amplitude in a Pulsed Column

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Abstract:
An experimental investigation was carried out initially to study the hydrodynamics of pulsed extraction column with nozzle plates. The experiments were conducted in a 50 mm diameter glass column with 26 standard nozzle plates having 23% free area. 30% TBP in Dodecane was used as continuous phase and 3 M Nitric acid as dispersed phase with organic to aqueous flow rates in the ratio of 3:1. Flooding curve was generated by varying pulsation conditions and throughputs. The experimental facility was then used to develop and validate a model-based online amplitude measurement methodology.

Introduction
Pulsed columns are extensively used in spent nuclear fuel reprocessing plants to separate, decontaminate and purify uranium and plutonium [1, 2]. The pulsed column used for solvent extraction is a vertical column with one contact zone containing a number of perforated horizontal plates, two enlarged ends for phase disengagement, one pulse leg for pulsing, and provisions for feeding and withdrawing aqueous and organic streams (see Fig. 1). The plate perforations are usually so small that substantial flows of aqueous and organic phases due to density difference do not occur. However, application of a periodic pulsation can provide additional energy required to force the liquids through the perforated plates and also to break up the liquids into small droplets and thereby, promoting high interfacial area and increased mass transfer.

In the pulse column design, the height of the contacting section is determined by the efficiency of mass transfer and the diameter of the column is determined by the flooding characteristics. Height of the pulse columns used in a nuclear reprocessing plant should be as small as possible in order to reduce the cost of the hot cell. Equipment design variables and operating variables together determine the efficiency and throughput of the pulsed column. The maximum throughput of the pulsed column is governed by flooding, which may be caused by either insufficient or excessive pulsing. Flooding is defined as the condition at which the two phases cannot pass counter-currently through the column resulting in at least one of the phases leaving from the same end of the column that it entered. This usually results in the rejection of the dispersed phase and the formation of a second interface at the opposite end from the dispersed phase outlet. The flow through the column is achieved from the energy provided by the pulsation. This energy input can be expressed as the product of the pulse amplitude ($a$) and the pulse frequency ($f$). This product ($af$) is used to correlate the extent of flooding in the column [3]. The minimum HTU (height of a transfer unit) values generally occur in an area between 75 and 95% of the flooding conditions [4]. Therefore, the flooding curve contains basic information for selecting the most favourable operating conditions for a given pulsed column.
In the present study, an experimental investigation was carried out initially to study the hydrodynamics of pulsed extraction column with nozzle plates. The experimental facility was used subsequently to develop and validate a model-based online amplitude measurement methodology.

The experimental setup is shown in Fig. 2. A pilot scale, glass pulsed extraction column with nozzle plates was used for all the experiments undertaken. The vertical glass pulsed column has a 1.5 m long contact zone with an inside diameter of 50 mm. The contact zone is formed by 26 stainless steel nozzle plates oriented downward and spaced 50 mm apart. Each plate is 1 mm thick and contains holes of 4.8/3.2 mm diameter and 1 mm indentation. The holes were laid out on a triangular pitch of 9.5 mm. The free area
was approximately 23% of the total area. The phase disengaging ends of the glass column are 600 mm long and have an inside diameter of 150 mm.

The air pulsing system comprises of an air compressor, a pressure regulating valve (PRV), a flow control valve (FCV), an air reservoir with a capacity of 500 L, a 3-way poppet type pulsing valve with electronic timer, a pulse leg and associated piping. The pulse leg is 2.5 m long and has an inside diameter of 25 mm. The PRV is provided to set the line pressure in the upstream side of the FCV, which regulates the compressed air flow to the reservoir and thereby maintains the tank pressure at a set value. The pulse column is provided with two pressure transducers, P1 and P2, to measure the differential pressure across the contact zone (see Fig. 2).

As shown in Fig. 2, the experimental facility is equipped with four stainless steel tanks (1000 L capacity each) for storing the feed and product streams and has provisions for pumping the organic (30% TBP in Dodecane) and aqueous (3M Nitric acid) feed streams to the pulsed column, and measuring their flow rates (F1 and F2). The organic product stream overflows from the column top disengaging end to the organic product tank. The aqueous product stream is withdrawn from the bottom disengaging end using a metering pump which maintains the bottom interface at the desired level.

**Experimental procedure**

The pulsed column was initially filled with 3M Nitric acid using the aqueous feed pump. Air pulsing was started under static column condition (without liquid feeds) to evaluate the performance of the air pulsing system. An initial static pulsing test was conducted without flow control valve to assess the effectiveness of pressure regulating valve (PRV) in regulating the air pressure in the reservoir. Test results indicated substantial fluctuations in the reservoir pressure which in turn resulted in unsteady pulsing of the liquid column. A flow control valve (FCV) with pressure feedback was introduced to regulate the compressed air flow and thereby, maintain the air reservoir pressure at the set value. Reservoir pressure transients with and without flow control valve are compared in Fig. 3, which illustrates that introduction of a flow control valve leads to better control on the air reservoir pressure and thus, stable pulsing of the column.

Dynamic pulsing experiments were carried out subsequently using 30% TBP in Dodecane as continuous phase and 3 M Nitric acid as dispersed phase with organic to aqueous flow rates in the ratio of 3:1. The pulsing frequency was maintained at 1 Hz in all experiments. The pulse amplitude was measured by observing the maximum displacement of liquid in the pulse leg with the help of a scale fixed on it. The corresponding amplitude in the actual column was calculated based on the difference in cross sectional areas of the leg and the column. The superficial velocities of organic (VC) and aqueous (VD) in the column were calculated based on the measured volumetric flow rates.

**Flooding curve and flow regimes**

Flooding curve for the glass pulsed column with nozzle plates was generated by varying the pulse amplitude for a given set of superficial velocities of the liquid feed streams. An O/A ratio of 3 was used in the present study. The pulse frequency was set at 1 Hz using an electronic timer and the pulse amplitude was regulated by adjusting the reservoir air pressure.
Flooding points corresponding to insufficient pulsing and excessive pulsing were obtained for different values of volumetric velocity (VC+VD). These points are presented as flooding curve in Fig. 4. When the volume velocity is 5 mm/s, flooding due to insufficient pulsing occurs at a pulse velocity, $a_f = 2.5$ mm/s and flooding due to excessive pulsing occurs at $a_f = 55$ mm/s. Point 1 in Fig. 4 corresponds to VC+VD = 5 mm/s and $a_f = 15$ mm/s. Fig. 5(1) presents the flow regime corresponding to Point 1 which shows that mixer-settler zone exists under this condition. It was observed that emulsion zone prevails when $a_f \geq 25$ mm/s. For example, Fig. 5(2) shows the emulsion flow regime corresponding to Point 2, for which VC+VD = 5 mm/s and $a_f = 30$ mm/s (see Fig. 4). Figs. 5(1) and 5(2) illustrate substantial enhancement in the interfacial mass transfer area at Point 2 compared to Point 1. Points 1 and 2 correspond to 27% and 60%, respectively, of the flooding $a_f$ for VC+VD = 5 mm/s under excessive pulsing condition.

Fig. 6 presents effect of pulse amplitude when the volume velocity is VC+VD = 10 mm/s while maintaining the pulse frequency at $f = 1$ Hz and O/A = 3. Panels (1), (2), (3) and (4) shown in Fig. 6 correspond, respectively, to Points 3, 4, 5 and 6 in Fig. 4. Flow visualization studies conducted confirm that mixer-settler flow regime exists when $a_f < 25$ mm/s and emulsion zone prevails when $a_f \geq 25$ mm/s. The experimental results establish that for a pulsation frequency, $f = 1$ Hz, emulsion flow regime occurs when the pulse amplitude, $a \geq 25$ mm. Thus, the present study ascertains the importance of on-line measurement of pulse amplitude to operate the pulse column in the most efficient flow regime (emulsion zone).

**On-line amplitude measurement in glass column**

The present experimental facility was used further to obtain a relation between the differential pressure across the contact zone and pulse amplitude in the column. Pulsing experiments were carried out using single phase flow under dynamic conditions for this
Two pressure transducers (Make: CEMAS, Model: U5156) were used to measure the differential pressure across the contact zone of the column and the data was recorded by a multi channel chartless recorder (Make: EUROTHERM, Model: 6100A). Fig. 7 shows the experimentally obtained differential pressure for pulsing amplitude of 1 inch in the 2 inch diameter glass column. It is observed from Fig. 7 that a low frequency component is superimposed over the high pulsing frequency. Therefore, a time-averaged differential pressure was obtained across the pulsed column based on the area under the differential pressure curve for the period corresponding to the low frequency component.

In order to establish the relation between the time-averaged differential pressure and the pulse amplitude in the column, different experiments were conducted by varying the pulse amplitude from 0.5 inch to 1.5 inch in the 2 inch diameter glass pulse column. The experimental results are presented in Fig. 8. It shows that the pulse amplitude in the 2 inch diameter glass column exceeds 1 inch when the time-averaged differential pressure across the contact zone is more than 2000 Pa. Thus, the time-averaged differential pressure can be used for the near-online calculation of the amplitude in the pulse column based on the amplitude-differential pressure relationship (see Fig. 8).

Fig. 6: Effect of amplitude for volume velocity of 10 mm/s (f = 1 Hz, O/A=3:1)
As it is difficult to generate the amplitude-differential pressure relationship for an industrial pulsed column experimentally, a model-based methodology was developed for the on-line measurement of pulse amplitude in the reprocessing plants. A mathematical model for the air pulsing can be used to generate the relation between the amplitude and differential pressure in the pulsed column. Such a model comprises of three sets of governing equations – first set of equations for describing the air behaviour during air supply to the pulse leg, second set for describing the air behaviour during air exhaust from the pulse leg and third set for describing the liquid behaviour in the pulse leg and the column. Formulation and solution of these governing equations are described in [5, 6, 7] and not described here. Fig. 9 compares the model predicted amplitude-differential pressure relationship with that obtained experimentally. It shows that the model predictions agree well with the experiments and the maximum error is about 10% when compared with the experiments using a 2 inch diameter glass column. Fig. 9 also shows a uniform under-prediction of the experimental results by the model. This is on account of the fact that the pressure transducers used in the experiments could record only 8 measurements per cycle, thereby,
resulted in a uniform under-prediction in the area-averaged differential pressure. Since purge method will be used for pressure measurement in the actual plant, the temporal variation in the differential pressure can be smooth and less noisy compared to that obtained using a pressure transducer. This will reduce the error between the measured values and the model predictions. In case of industrial columns, the mathematical model needs to be tuned based on the static pulsing experiments during commissioning. The tuned model can be used to generate a calibration curve for calculating the pulse amplitude in the column based on online measurement of the differential pressure across the contact zone.

Conclusions

The present study establishes the importance of online measurement of pulse amplitude to operate the pulse column in the most efficient flow regime. The experimental results shows that a pulse amplitude of $a \geq 25$ mm is required to maintain emulsion flow regime for a pulsation frequency of $f = 1$ Hz. From the experimental data, it is ascertained that the time-averaged differential pressure can be used for the near-on-line calculation of the amplitude in the pulse column based on the amplitude-differential pressure relationship. This study also illustrates that a mathematical model for the air pulsing can be used to generate the relation between the amplitude and differential pressure in the column. The mathematical model can be tuned based on the static pulsing experiments during commissioning. The tuned model can be used to generate a calibration curve for calculating the pulse amplitude in the column based on online measurement of the differential pressure in the plant.

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