Proc. Pakistan Acad. Sci. 44(3):189-195.2007

EFFECT OF OPERATING PARAMETERS ON EXTRACTION IN SIEVE PLATE PULSED EXTRACTION COLUMN

Syed Sheraz Daood*, Shahid Munir, Niaz Ahmed Akhtar and M. Arif Butt

Faculty of Engineering and Technology, University of the Punjab, Quaid-e-Azam Campus, Lahore (54590), Pakistan

Received March 2006, accepted August 2007

Communicated by Prof. Dr. Abdul Raouf

Abstract: In the present study the effect of pulsation frequency, stroke length, continuous phase and dispersed phase flow rates were investigated on mass transfer characteristics i.e. percentage of solute in extract phase. Experiments were carried out on vertically installed sieve plate Pulsed Extraction Column by taking acetic acid-water mixture and ethyl acetate as feed solution and solvents, respectively. Pulsed column was 365 cm tall and of 5 cm internal diameter. Experimental results indicated that an increase in the pulsation frequency from 25 to 112 strokes/min resulted in substantial decrease in the time required to attain steady state for percentage of solute in extract phase. Steady state value for concentration of solute in extract phase was obtained with 67 percent reduction in time for higher value of pulsation frequency. It was observed that increase in the concentration of solute in extract phase was remarkable for values of pulsation frequency from 10 to 35 strokes/min as compared to higher values of pulsation frequency. It was also confirmed that stroke length acts as driving force towards mass transfer of solute in extract phase and by increasing stroke length form 0 to 15 mm, the percentage of acetic acid in extract phase increased from 6.2% to 7%. Effect of variation of dispersed phase and continuous phase flow rates on concentration of solute in extract phase was also studied.

Keywords: Pulsation frequency, steady state, dispersed phase, extract phase, percentage extraction, stroke length, mass transfer characteristics

Introduction

Solvent extraction is a technique used for separating components in solution by distribution between two immiscible liquid phases [1]. Solvent extraction is one of the most widely used unit operations involved in process industry. This mass transfer operation consists in separating one or several substances (solute) present in a solid or a liquid phase by contacting it with another liquid phase in which these substances are preferentially transferred [2].

In Liquid-Liquid extraction operation, a liquid solution (the feed) is contacted with an immiscible or nearly immiscible liquid (solvent) that exhibits preferential affinity towards one or more of the components in the feed. The added solvent is also soluble with a specific solute contained in the solution [3]. Among many other important characteristics, the basic strong ones are distribution coefficient, minimum solubility in raffinate phase, physical properties like high interfacial tension (which promotes phase separation and low viscosity promoting both mass transfer and phase separation), effect of losses in raffinate, flammability, availability and cost.

There has been scarce information published on the study of system parameters such as pulsation frequency, effect of flow rates on

Corresponding author: Syed Sheraz Daood
Lecturer, Institute of Chemical Engineering & Technology, Faculty of Engineering and Technology
University of the Punjab, Quaid-e-Azam Campus, Lahore (54590), Pakistan
Email: daood_sheraz@yahoo.com

the steady state recovery of the solute and percentage saving in time. However, Vatanatham *et al.* [4] studied HTU of acetone-toluene-water extraction in a pulsed column. The mass transfer correlation in a pulsed column, particularly the HTU, is needed for column design. They carried out the study on the HTU of pulsed perforated plate tower for liquid-liquid extraction. In the experimental ternary system, water extraction solvent was used to extract acetone solute from toluene feed solvent in a counter-current extraction scheme. Operating variables such as pulsing frequency and amplitude, solute concentration, drop diameter, and flow rate of heavy liquid and light solution were investigated. Some work has also been done by Yasin *et al.* [5] for developing differential property balance, continuous phase mass transfer and dispersed phase mass transfer equations along with modification in general differential property balance for drop population balance.

Additional work in this area of research involves numerical studies for flow and heat transfer characteristics of a fully-developed pulsating flow in a strongly curved pipe, where emphasis is placed on delineating the effects of the Reynolds number, and pulsation amplitude and frequency. Chung *et al.* [6] developed the time-dependent incompressible Navier-Stokes equations. Particular attention is given to heat transfer properties over substantially extended parameter ranges of the Reynolds number *Re* and the Womersley number *Wo*. Use is made of a well-established numerical solution procedure, with minor amendments. The computed results on the flow field are in close agreement with the existing data in the overlapping parameter ranges. The spatial distributions of axial and secondary flows are depicted. When *Wo* is small, the time and space-averaged Nusselt number is lower for a pulsating flow than for a corresponding non-pulsating flow. At moderate and high *Wo*, however, the difference in between a pulsating and a non-pulsating flow is insignificant.

Similarly Moschandreou *et al.* [7] determined that how pulsation affects the rate of heat transfer and how the phenomenon depends on the Prandtl number and on pulsation frequency. The results indicate that in a range of moderate values of the frequency there is a positive peak in the effect of pulsation whereby the bulk temperature of the fluid and the Nusselt number are increased, but the effect is reversed when the frequency is outside this range. The peaks are higher at lower Prandtl numbers. Kim *et al.* [8] also carried out experimental study on heat transfer in the thermally developing region of a pulsating channel flow.

In liquid-liquid extraction operation two streams result from the contact of feed and solvent; the extract, which is the solvent rich solution containing the desired extracted solute, and the raffinate, the residual feed solution containing little solute. The technique of solvent extraction finds its applications in chemical and pharmaceutical industry [9].

In general, there are many examples where liquid-liquid extraction supercedes the role of distillation, expensive evaporation, fractional crystallization and separation of close boiling liquids. Acetic acid manufacturing, removal of phenol from wastewater, recovery of caprolactam from ammonium sulphate solutions, extraction of tetraethyl lead from brine, removal of mercaptans from naphtha [10], penicillin extraction and many more can be quoted as further examples.

In liquid-liquid extraction there are many types of equipments which are used on laboratory and industrial scale [11]. Over the years different types of solvent extraction contactors have been developed for use in the mineral process industry. The most common type is the mixer settler arrangements but recently pulsed columns have been employed because they act as small foot print for multistage extract systems [1]. These pulsed columns found their rapid use in nuclear industry due to their minimum mechanical maintenance during continuous operation [12]. The pulsed column was first applied to uranium extraction by Groot which helped in achieving more than two fold reduction in the column height from the heights needed with a conventional packed column [5].

In the present study, pulsation column was used because of its low maintenance cost and effective mass transfer characteristics. Ethyl acetate was used as a solvent because of its relatively greater affinity, interfacial tension, low viscosity and minimum solubility in raffinate phase as shown in the following Table 1.

Table 1
Solvents for Acetic Acid Extraction [13].

Solvent	Distribution Coefficient at 20^o C
Ethyl acetate	0.9
MIBK	0.7
Toluene	0.06
n-Hexane	0.01

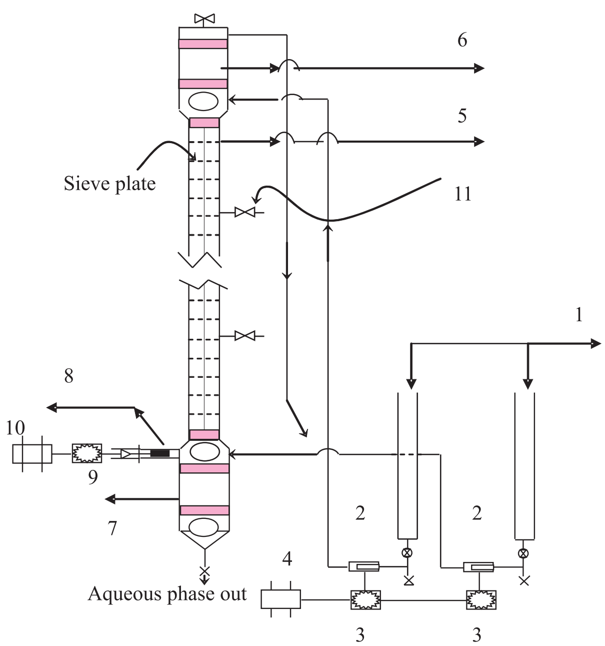
Materials and Methods

The Sieve Plate Pulsed Extraction Column (pilot plant scale) used in this study is shown in Fig 1.

The column consisted of three sections; extraction section (7.167 L hold up) consisted of 5 cm internal diameter glass tube having a height of 365 cm, containing 74 sieved plates of 5 cm dia each having 32 holes of 3.175 mm dia, dispersed and continuous phase settling sections laying above and below the extraction phase, respectively, each having 50 cm height and 4.166 L hold up.

Feed solution (acetic acid and water) and solvent (ethyl acetate) were introduced from the bottom and top of the column, respectively, with the help of two metering pumps. These metering pumps ran through a single motor but each had a separate speed reduction gear box so that flow rates of solvent and feed solution could be adjusted independently. There were two feed cylinders for injection of solvent and feed mixture to these pumps, each having a volume of 1.527 L.

Pulsation through the system was introduced by reciprocating type piston pump. The frequency of the pulsation could be changed with help of a gear box system and there was also provision for changing the pulsation stroke or amplitude (stroke length). To protect the apparatus from damage due to very high pulsation speed, the dial for controlling the pulsation frequency was installed with a safety knob so that pulsation frequency could not be increased above a safe limit. These accessories were used to study



1. Feed Cylinder
2. Feed Pump
3. Gear Box
4. Feed Pump Motor
5. Extraction Section
6. Dispersed Phase Settling Section
7. Continuous Phase Settling Section
8. Pulsation Pump
9. Pulsation Pump Gear Box
10. Pulsation Pump Motor
11. Sampling Points

Figure 1. Schematic Diagram of Pilot Plant Scale, Sieve Plate Pulsed Extraction Column.

the effect of various values of pulsation frequency and stroke length on percentage of solute in extract phase.

Before performing the actual experimental work both the injection metering pump and pulsation pump were calibrated for percentage opening versus volumetric flow rate, dial reading versus pulsation frequency, respectively. After the calibration of injection metering pump and pulsation pump, experimental work was carried out to study the effects of variation of pulsation frequency on time to achieve steady state for concentration of solute in extract phase. The effects of stroke lengths, dispersed phase and continuous phase flow rates on concentration of solute in the extract phase were also studied.

Results and Discussion

The effect of pulsation frequency on time to achieve steady state value of percentage of solute was studied while keeping the respective flow rates of feed mixture and solvent constant. The results obtained are shown in Fig. 2 and support the fact that with increase in the pulsation frequency the time required to attain steady state for percentage of solute in extract phase decreased substantially.

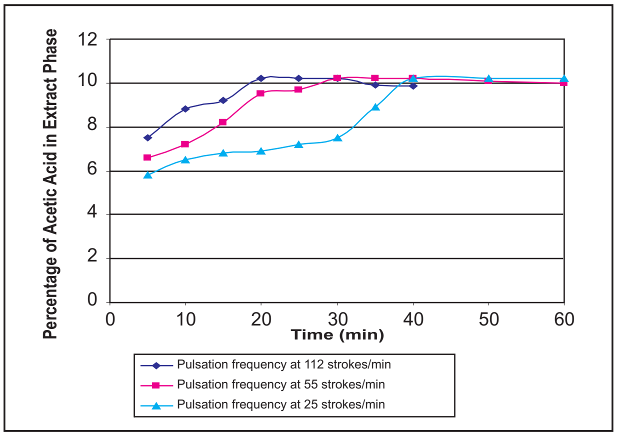


Figure 2. Effect of Pulsation Frequency on Time and Percentage of Solute in Extract Phase.

The required steady state value of extraction (10.2%) was obtained in just 20 min resulting in 67% reduction in time for higher value of pulsation frequency i.e., at 112 strokes/min. The results are also presented in Fig 3.

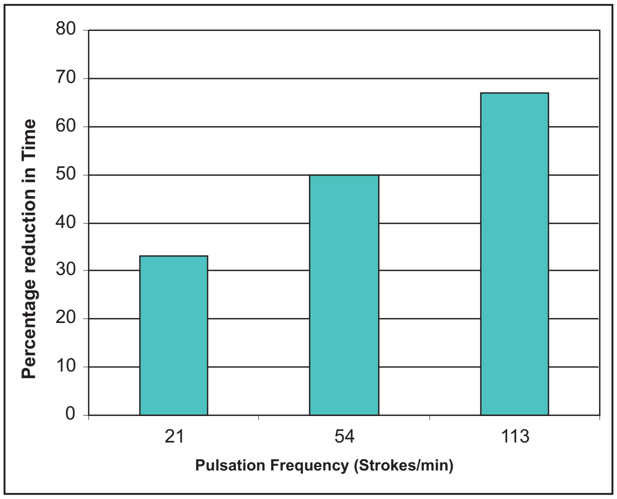


Figure 3. Bar Chart between Pulsation Frequency and Percentage Reduction in Time.

Increase in pulsation frequency resulted in appreciable decrease in time to achieve steady state value of percentage of solute in the extract phase as shown in Figs. 4 and 5. Higher values of pulsation frequency resulted in breakup of drops of dispersed phase which in return enhanced the mass transfer of solute in solvent. Krasuk *et al.* [14] proved that an increase in both frequency and amplitude of the pulse improves

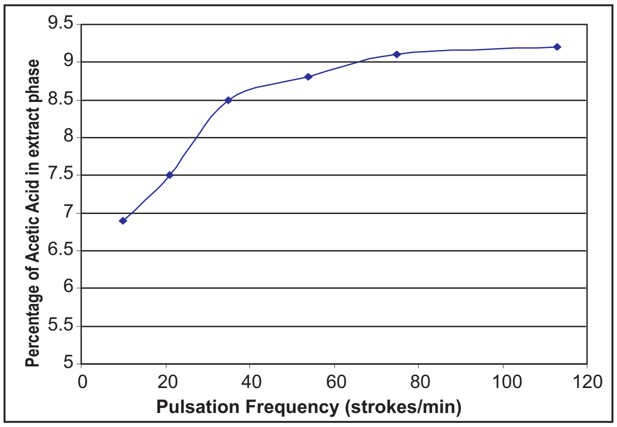


Figure 4. Effect of Pulsation Frequency on Percentage of Solute in Extract Phase.

the mass transfer coefficient. The increase due to pulsation is significant at all Reynolds number (N_{Re}) in the streamline flow region. The results found are in conformity with those reported by Krasuk *et al*. though obtained with a different system.

The effect of pulsation frequency on the percentage of solute (acetic acid) in extract phase while keeping the time of operation constant was studied (Fig 4). For this purpose, several runs were carried out. The dispersed phase flow rate (mixture of acetic acid and water) and continuous phase flow rate (solvent i.e. ethyl acetate) were kept at equal values. Fig. 4 shows that with the increase in pulsation frequency, the percentage of acetic acid in the extract phase increased with the time of operation and constant feed flow rates. With the increase in pulsation frequency from 10 to 35 strokes/min, the percentage of acetic acid in extract phase showed a steeper rise from 6.9% to 8.5%. However, later values indicated that the profile was dampened down and thus the optimum value of pulsation frequency to attain the maximum percentage of solute in a pulsed column was determined. The dampened part of the graph is attributed to the formation of much smaller droplets of dispersed phase which behave like solid spheres resulting in inhibition of mass transfer of solute in the solvent as reported by Swati e*t al*. [15].

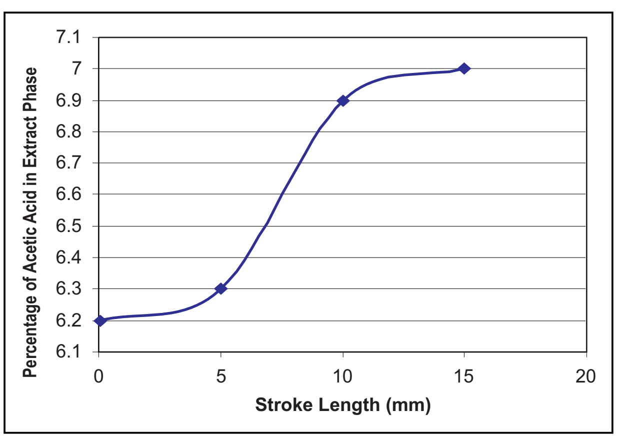


Figure 5. Effect of Stroke Length on Percentage of Solute in Extract Phase.

In performing the above mentioned set of test runs, the effect of different stroke lengths on the percentage of acetic acid in the extract phase was also studied and the trend shown in Fig. 5 was obtained at the pulsation frequency of 21 strokes/min, while flow rates of both the feed mixture and solvent were kept constant. The percentage of acetic acid in extract phase increased from 6.2% to 7% with the increase in stroke length from 0 to 15 mm. The trend endorses the fact that stroke length acts as a driving force which in turn increases the turbulence and results in more mass transfer of the percentage of the solute (acetic acid) in the extract phase. Krasuk *et al.* [14] also discussed the same parameter as amplitude and proved that increase in value of amplitude increases the mass transfer coefficient.

To see the effect of variation of dispersed phase flow rate on the percentage of acetic acid in the extract phase, a few runs were carried at fixed continuous phase flow rate and pulsation frequency of 55 strokes/min. The results obtained are presented in Fig. 6

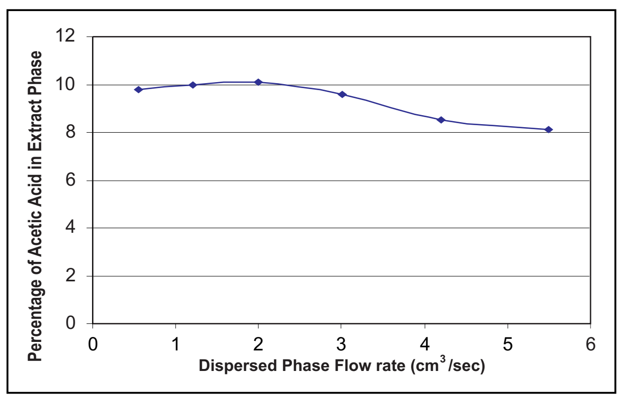


Figure 6. Effect of Dispersed Phase Flow Rate on Percentage of Solute in Extract Phase.

Similarly, the effect of the variation of continuous phase flow rate on the percentage of acetic acid in the extract phase was also studied at fixed pulsation frequency of 55 strokes/min and dispersed phase flow rate at 50% pump opening. The results obtained are plotted as percentage of acetic acid in the extract phase versus continuous phase flow rate in Figure 7.

Fig. 6 shows that there is a decreasing trend between dispersed phase flow rate and percentage of acetic acid in the extract phase, while a reverse behavior is seen in case of variation of continuous phase flow rate and percentage of acetic acid in the extract phase as in Fig. 7. The increase in the flow rate of continuous phase supported the fact that a greater percentage of acetic acid was extracted in the extract phase because of additional amount of solvent running into the system. However, in the case of variation of dispersed phase flow rate, the percentage of solute in the extract phase increased initially before decreasing later because of axial mixing. Bardin-Monnier *et al.* [2] reported that axial mixing takes place within the column and reduces the efficiency of the process by decreasing solute concentration gradients and as a consequence the mass transfer rate decreases. For this reason, this axial mixing has to be reduced at its lowest level and has to be taken into account in the design of industrial columns. Neglecting the axial mixing phenomena inevitably leads to an over evaluation of the process efficiency.

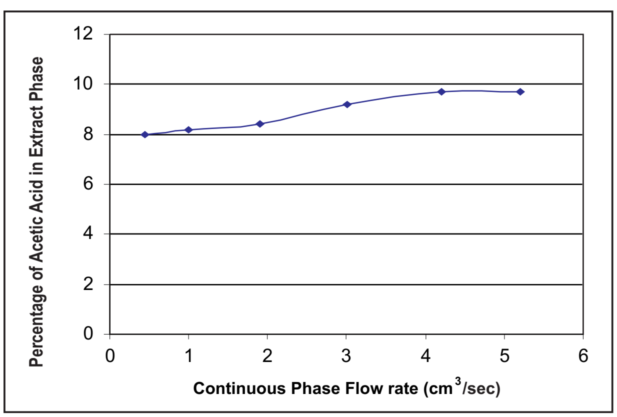


Figure 7. Effect of Continuous Phase Flow Rate on Percentage of Solute in Extract Phase.

Conclusion

Increase in pulsation frequency resulted in appreciable reduction in time to achieve steady state value of percentage of solute in extract phase as shown in Figures 2 and 3. For higher values of pulsation frequency, the breakup of drops of dispersed phase was obvious which greatly enhanced the mass transfer. However, beyond the optimum value of pulsation frequency as mentioned in Figure 4, formation of much smaller droplets of dispersed phase took place. These much smaller droplets behaved like solid spheres which started to inhibit the mass transfer of solute in solvent.

The trend shown in Figure 5 confirms that stroke length acts in support of mass transfer of percentage of solute in extract phase because it works as driving force which in turn increases the turbulence. In fact, by increasing stroke length from 0 to 15 mm the percentage of acetic acid in extract phase increased from 6.2% to 7%.

From Figure 6, it is clear that with the increase in dispersed phase flow rate at constant continuous phase flow, the percentage of solute in the extract phase increased initially and then decreased because of axial mixing. As axial mixing within the column reduces the efficiency of the process by decreasing solute concentration gradients, the mass transfer rate decreases as a consequence. Whereas with increase in flow rate of continuous phase for fixed dispersed phase flow rate, the percentage of solute in the extract phase continued to increase.

References

1. **Bujalski, J.M., Yang, W., Nikolov, J., Solnordal, C.B. and Schwarz, M.P.** 2006. Measurement and CFD simulation of single phase flow in solvent extraction pulsed column. *Chem. Eng. Sci*. 61:2930-2938.
2. **Bardin-Monnier, N., Guiraud, P. and Gourdon, C.** 2003. Residence time distribution of droplets within discs and doughnuts pulsed extraction columns via Langrangian experiments and simulations. *Chem. Eng. J.* 94:241-254.
3. **Hanson, C. and Murthy, S.L.N.** 1974. Recovery of magnesium chloride from sea water concentrates.

Proc. Int. Solvent Extr. Confr. - ISEC. 1: 779-790.

4. **Vatanatham, T., Terasukaporn, P. and Lorpongpaiboon P.** 2003. HTU of acetone-toluene-water extraction in a Pulsed Column. *Kasetsart University Publication Index*, 393-394.
5. **Yassin, S., Aslam, N., Ahmad, N. and Butt, A.** 1999. Mathematical modeling of a pulsed extraction column. *J. Pak. Inst. Chem. Eng.* Vol. XXVIII, 29-36.
6. **Chung, J.H. and Hyun, J.M.** 1994. Heat transfer from a fully-developed pulsating flow in a curved pipe. *Int. J. Heat Mass Transfer* 37:43-52.
7. **Moschandreou, T. and Zamir, M.** 1997. Heat transfer in a tube with pulsating flow and constant heat flux. *Int. J. Heat Mass Transfer* 40:2461-2466.
8. **Kim, S.Y., Kang, B.H. and Hyun, J.M.** 1993. Heat transfer in thermally developing region of a pulsating channel flow. *Int. J. Heat Mass Transfer* 36:4257-4266.
9. **Coulson, J.M. and Richardson, J.F.** 2003. *Chemical Engineering*, Vol. 2, 4th ed., Butterworth Heinemann. London.
10. **Bailes, P.J.** 1977. Solvent extraction in the petroleum and petrochemical industries. *Chem. Ind.* 69–73.
11. **Godfrey, J.C. and Slater, M.J.** 1994. *Liquid-liquid extraction equipment*, John Wiley & Sons.
12. **Rouyer, H., Lebouhellec, J., Henry, E. and Michel, P.** 1974. Present study of extraction pulsed columns, *Proc. Int. Solvent Extraction Confr. – ISEC.* 74:2339-2353.
13. **Perry, R.H. and Green, D.** 1997. *Perry's Chemical Engineering Hand Book*. 7th ed., McGraw Hill.
14. **Krasuk, J.H. and Smith, J.M.** 1963. Mass transfer in a pulsed column. *Chem. Eng. Sci.* 18:591-598.
15. **Swati, M. and Vogelpohl, A**. 1997. A simplified hydrodynamic model for a pulsed sieve plate extraction column. *Chem. Eng .Process.* 36:385-395.