# Investigation of the effects of tray downcomer design on the efficiency of a vibrating plate extractor

Nomakhosi D. Sincuba, Sudesh Rathilal, Milan Carsky<sup>\*</sup> Department of Chemical Engineering, Durban University of Technology, 79 Steve Biko Road, PO Box 1334, Durban, South Africa Corresponding author: Sudesh Rathilal, Email address: <u>rathilals@dut.ac.za</u>

## Abstract

The effectiveness of different tray designs was studied in liquid-liquid extraction using a vibrating plate extraction (VPE) column. The effect of these tray designs and agitation levels on the dispersed phase holdup, drop size distribution and Sauter mean diameter was investigated. Two different sieve tray designs (Type 1 – cylindrical downcomers and Type 2 – straight segment downcomer) were tested. It was found that the Type 1 was more efficient than Type 2. It had a higher dispersed phase holdup and lower Sauter mean diameter with the highest percentage of solute being extracted (95.05%).

## 1. Introduction

Liquid-liquid extraction is a separation process that involves the mass transfer of a solute from a liquid mixture to an immiscible liquid solvent. Liquid-liquid extraction is also known as solvent extraction. It is ranked as the second most vital separation process (the first being distillation) that is extensively applied by industries (Mohanty and Volgelgohl, 1997). Solvent extraction is normally used when distillation is impractical because of the close relative volatilities of the two components and when there is a possibility of azeotropes being formed (Seader and Henley, 2006; Humphrey and Keller, 1997). It is used by several industries for various applications such as production of active agents by the pharmaceutical industry, production of monomers and aromatics by the petroleum industry, and for cleaning of waste water to separate dissolved compounds (Mohanty, 2000). Liquid-liquid extraction uses various devices such as the application of gravitational forces or centrifugal forces for separation purposes and can be mechanically agitated in order to improve interfacial area and mass transfer (Seader and Henley, 2006; Rama Rao et al., 1991). There are various types of agitated columns and the vibrating plate extractor is one type of mechanically agitated column (Rathilal et al., 2011). It is also a modification of the Karr reciprocating plate column (RPC) (Seader and Henley, 2006). The main difference between the two types lies in the design and function of the tray. In the Karr reciprocating plate column the tray is open structure with large holes and large free area (approximately 58%) while in the vibrating plate column, the trays have small perforations and a small free area with

<sup>\*</sup>Professor Emeritus, University of KwaZulu-Natal, School of Engineering, Durban, South Africa

or without downcomers (Aravamudan and Baird, 1999). The small perforations provide passage for the dispersed phase, while one or more downcomer(s) on each plate provide passage for the continuous phase. The downcomers allow for a much higher throughput than would be possible using perforations alone (Lo et al., 1992).

The advantage of VPE over RPC is that the RPC always operates in the emulsion regime while the VPE can operate in a mixer-settler, dispersion or emulsion regime (Shen et al., 1985; Lo and Prochazka, 1983). Thus Karr columns can only operate at high frequencies whereas the vibrating plate extraction column can operate at low and high frequencies. It also implies that a vibrating plate extraction column uses less energy than a Karr column in order to achieve the same throughput. Apart from the column diameter, column height, weir height, perforation size and tray spacing the vibrating plate extraction column has many parameters that can be adjusted to achieve the desired results such as types of trays and frequency and amplitude of vibrations in order to improve its mass transfer. These parameters can affect both the hydrodynamics and mass transfer of the system. Much more research work has been done in enhancing the performance of the Karr reciprocating plate column (Kumar and Hartland, 1988; Aleksic et al., 2002) as compared to the vibrating plate extraction column.

Most of the work that has been done on the VPE focused on hydrodynamics (Aravamudan and Baird, 1999; Rama Rao et al., 1991; Nemecek and Prochazka, 1974) and mass transfer effects on the hydrodynamic conditions of the vibrating system is significant in designing the column since it affects both the drop size diameter and dispersed phase holdup. The main objective of this study is to investigate the effect of tray design (downcomer type) in order to enhance the effectiveness of liquid-liquid extraction. The results show an investigation of the effects of the downcomer type on the distribution in terms of Sauter mean diameter and dispersed phase holdup with and without mass transfer and also the effects of agitation level (product of amplitude and frequency of vibrations) on the extent of mass transfer.

#### 2. Experimental

#### 2.1 System used

The acetone-toluene-water system was used in this study as a standard test system for liquid extraction as recommended by the European Federation for Chemical Engineering, (EFCE, 1985). This system was used because it has an advantage of having high accuracy and repeatability (Saien and Daliri, 2008). Technical grade acetone and toluene was used in this study without any further purification. Tap water was used as a solvent to extract acetone from toluene. The experiments were conducted at ambient conditions.

#### **2.2 Experiment Procedure**

The phases were pumped to a column by positive displacement peristaltic pumps with the heavy aqueous continuous phase flowing down and the organic dispersed phase flowing up the column. Due to the fluctuations of the rotameters caused by the pumps and the vibrating tray, surge tanks were used to prevent these pulses from being transmitted to the rotameters. The glass column was constructed of sections of 47.7 mm internal diameter tubes with an active height of 4.7 m. The plate perforations were 2.98 mm holes and two types of downcomers were used. Type 1 and Type 2 trays are shown in Figure 1 and Figure 2 respectively.



Figure 1 – Type 1 (cylindrical downcomer)



Figure 2 – Type 2 (straight downcomer)

Besides the downcomer type, the trays were similar in all other aspects. The trays were spaced at 150 mm apart. The interface was located at the top settling tank due to the fact that the dispersed phase was the light phase (Humphrey and Keller, 1997). A conductivity probe linked to the extract pump was used to maintain the interface level which was set at a fixed measured level below the raffinate overflow point. The column was initially filled with the continuous phase. Dispersed droplets were allowed to rise through the column and through perforations of the plates. The plate stack was vibrated using a vibrating motor, attached to the centre shaft of the plate stack, and was located at the top of the column. The system was allowed to run for 45 minutes to reach steady state (Rathilal, 2010). The agitation level was set to the required value by adjusting the frequency on the vibration motor controller. The effect of agitation level (product of amplitude and frequency of vibration) on hydrodynamics and on mass transfer was done by changing the frequency from 0.5 Hz to 3 Hz in increments of 0.5 Hz whilst the amplitude remained fixed at 2.5 mm. A Perspex box filled with water was placed around the column to prevent the effect of the curvature of the column during photography of the droplets to get the correct size of the droplets. The drop sizes measured using photographic techniques were evaluated with the aid of Image Pro Plus

software and then the Sauter mean diameter  $(d_{32})$  was calculated from the drop size distributions using the following formula:

$$d_{32} = \frac{\sum n_i d_i^{3}}{\sum n_i d_i^{2}}$$
(1)

The dispersed phase holdup was measured by taking the difference of the interface level before and after stopping the feed pumps of both phases and after the dispersed phase droplets were allowed to coalesce and accumulate in the top settling tank. Dispersed phase holdup was estimated using the following formula:

$$Dispersed \ phase \ holdup = \frac{active \ volume \ of \ the \ dispersed \ phase}{active \ volume \ of \ the \ column}$$
(2)

The percentage amount of acetone extracted was measured by analysing the feed sample and the raffinate sample using gas chromatography. The percentage amount of acetone extracted was estimated using the following formula:

Percentage amount of acetone extracted = 
$$\frac{x_f - x_r}{x_f} \ge 100$$
 (3)

Where  $x_f$  and  $x_r$  are the mass fractions of acetone in the feed and in the raffinate respectively.

#### **3. Results and Discussion**

# **3.1** Effect of mass transfer on the dispersed phase holdup and Sauter mean diameter

The mass transfer experiments were conducted using the acetone-toluene-water test system, with the feed consisting of 6% solute (acetone). The dispersed phase holdup, Sauter mean diameter and drop size distribution were determined for a S/F ratio of 1:1 and for each tray design (tray with circular downcomers and tray with a straight segment downcomer) for various agitation levels. A repeatability analysis for mass transfer experiments (for Type 1 tray) was conducted in order to find the ideal regime.

The experimental results are divided into two parts. The first part shows the effect of mass transfer on the hydrodynamic characteristics (dispersed phase holdup and Sauter mean diameter) using Type 1 tray. The second part shows the effect of both tray designs on the dispersed phase holdup as well as on Sauter mean diameter.

The effect of mass transfer on the dispersed phase holdup is shown in Figure 3 and the effect on Sauter mean diameter is shown in Figure 4. All results indicated are average values of at least 3 experimental runs.



Figure 3 – Effect of mass transfer on dispersed phase holdup using Type 1 tray

The agitation level ranged between 1 mm/s to 7.5 mm/s. Figure 3 shows higher dispersed phase holdup values in the absence of mass transfer. This indicates that the dispersed phase holdup without mass transfer cannot be used in designing the extraction column since it is affected by mass transfer. Figure 3 also indicates two distinct hydrodynamic flow regimes. The first flow regime is the mixer-settler regime where initially a high dispersed phase holdup was observed. This was attributed to the accumulation of a toluene layer under each tray in the column. Subsequently, as the agitation level was increased, this accumulated layer decreases, which corresponds to a decrease in the holdup. The smallest dispersed phase holdup, which shows the transition from the mixer-settler flow regime to the dispersion regime, was observed at 3.375 mm/s for both trends. Similar trends were observed in other published work during the transition period (Rathilal et al., 2011; Aravamudan and Baird, 1999).

As the agitation level was increased after the minimum holdup was reached, the dispersed phase holdup increased. An Increase in the agitation level caused the plate to vibrate faster which resulted in the formation of smaller droplets. The drag force acting on the droplets increased in relation to the buoyancy therefore the velocity of the drops decreased resulting in a higher residence time which caused a higher holdup (Camurdan, 1986; Taylor et al., 1982).

Additionally, for any further increase in the agitation level in the dispersion regime, an exponential increase in the dispersed phase holdup is expected, which drives the system to the emulsion flow regime. In this regime, intensive agitation levels resulted in the

reduction in the coalescence of the droplets. Thus the rate of entry of the dispersed phase droplets exceeded the rate of coalescence of the droplets which resulted in the flooding of the column (Laddha and Degaleesan, 1983). Therefore it was assumed that the emulsion regime was not favoured since the holdup increased in an unstable manner because of flooding. According to Aravamudan and Baird (1999), the dispersed phase holdup is a key factor in determining the interfacial area for mass transfer as well as an indication of the onset of flooding.

The effect of mass transfer on the Sauter mean diameter is shown in Figure 4 where the Sauter mean diameters with mass transfer were found to be higher than the Sauter mean diameters without mass transfer (although only slightly).



Figure 4 – Effect of mass transfer on the Sauter mean diameter using Type 1 tray

The mean drop diameters were normally larger when there was mass transfer. This was due to the enhanced coalescence effect during mass transfer as a result of the formation of larger droplets. This shows that mass transfer cannot be predicted using values of Sauter mean diameter in the absence of mass transfer as the values are affected by mass transfer. It is evident that there is a decrease in the Sauter mean diameter as the agitation level increases for both trends (with and without mass transfer). This result is expected since at higher agitation levels, the plates vibrated much faster, dissipating more energy to the fluid, producing much smaller droplets. It was also noted that at higher agitation levels, the inertial and shear forces of the droplets increased which as a result hinders the coalescence of the droplets hence resulting in smaller size droplets and a smaller Sauter mean diameter (Lo and Prochazka, 1983).

# 3.2 Effect of tray design on the dispersed phase holdup and Sauter mean diameter

Experimental results, for different tray designs were compared to determine whether the dispersed phase holdup was affected by the type of downcomer used in the vibrating plate extractor. The comparison of the dispersed phase holdup between the two tray designs is displayed in Figure 5.



Figure 5 – Effect of different tray designs on the dispersed phase holdup

Slightly higher values of dispersed phase holdup were obtained with the circular downcomers than with the straight segment downcomer. It appears that the tray with circular downcomers have a more effective dispersion action than the tray with the straight segment downcomer. Thus a multi-pass flow of the continuous phase resulted in an increased interfacial area.

Drop size distribution results were used to determine the Sauter mean diameter. The Sauter mean diameter was established for each tray design at a S/F ratio of 1:1. The effect of tray design on the Sauter mean diameter is shown in Figure 6.



Figure 6 – Effect of different tray designs on the Sauter mean diameter

Illustrated in figure 6 is a decrease in Sauter mean diameter as the agitation level increases. Figure 6 shows no significant effect of downcomer type on the Sauter mean diameter, which means that Sauter mean diameter is independent of the downcomer used.

#### **3.3 Effect of agitation Level on the mass transfer efficiency**

The percentage of acetone extracted was calculated using the feed and raffinate acetone mass fractions (equation 3). The effect of tray design on the percentage of acetone extracted is shown in Figure 7.



Figure 7 – Effect of tray design on the percentage of acetone extracted

The percentage of acetone extracted, as illustrated in figure 7, shows an increase in the amount of acetone extracted as the agitation level increases. The results also show a

significant increase in the amount of acetone extracted when the tray with circular downcomers is used (especially in the mixer-settler regime). The interfacial area available for mass transfer plays an essential role as well in the extraction effectiveness of a vibrating plate extraction column. Aravamudan and Baird (1999) showed that the interfacial area depends on the dispersed phase holdup and the Sauter mean diameter. Therefore with a larger dispersed phase holdup and a lower Sauter mean diameter, a larger interfacial area is available for mass transfer and therefore there is larger extent of extraction of acetone.

#### 4. Conclusion

There was no significant effect of tray design on the Sauter mean diameter. The results show a significant effect of tray design on the dispersed phase holdup as well as on the percentage amount of acetone extracted. It was found that Type 1 tray was more efficient than the Type 2 tray design. Type 1 tray shows high dispersed phase holdup values and low Sauter mean diameter values with the highest percentage amount of acetone extracted (95.05%).

## 5. References

- Aleksic, M., Veljkovic, V.B., Bankovic-Ilic, I., Lazic, M.L., Skala, D.U. 2002. Gas holdup in a gassed reciprocating plate column with raschig rings placed in interplate spaces. *The Canadian Journal of Chemical Engineering*. 80: 485-490.
- Aravamudan, K., Baird, M.H.I. 1999. Effect of mass transfer on the hydrodynamic behavior of a Karr reciprocating plate column. *Industrial and Engineering Chemistry Research*. 38: 1596-1604.
- Camurdan, M.C. 1986. Application of adaptive control to a reciprocating plate liquidliquid solvent extraction column. *Open Access Dissertations and Theses*, Paper 1068, Canada.
- EFCE, European Federation of Chemical Engineering. 1985. *Standard test system for liquid extraction*. 2<sup>nd</sup> Edition, E Publication Series: 46
- Humphrey, J.L., Keller, G.E. 1997. *Separation process technology*, McGraw-Hill, Chapter 3: 113-151.
- Kumar, A., Hartland, S. 1988. Prediction of dispersed phase holdup and flooding velocities in Karr reciprocating plate extraction columns. *Industrial and Engineering Chemistry Research*. 27: 131-138.
- Laddha, G.S., Degaleesan, T.E. 1983. Dispersion and coalescence. In *Handbook of Solvent Extraction*. Editors: Lo, T.C., Baird, M.H.I., Hanson, John Wiley & Sons, Chapter 4.
- Lo, T.C., Prochazka, J. 1983. Reciprocating-plate extraction columns. In *Handbook of Solvent Extraction*. Editors: Lo, T.C., Baird, M.H.I., Hanson, 372-389.

- Lo, T.C., Baird, M.H.I., Rama Rao, N.V. 1992. The reciprocating plate column development and applications, *Chemical Engineering Communications*: 116: 67-88.
- Mohanty, S., Volgelpohl, A. 1997. A simplified hydrodynamic model for a pulsed sieve plate extraction column. *Chemical Engineering Process*. 33: 385-395.
- Mohanty, S. 2000. Modelling of liquid-liquid extraction column: A review. 199-248.
- Nemecek, M., Prochazka, J. 1974. Longitudinal mixing in a vibrating-sieve-plate column. *The Canadian Journal of Chemical Engineering*. 52: 739-749.
- Rama Rao, N.V., Vijayan, S., Baird, M.H.I. 1991. Hydrodynamics of a vibrating perforated plate extraction column. *The Canadian Journal of Chemical Engineering*. 69: 213-221.
- Rathilal. S. 2010. Modelling of a vibrating plate extraction column. *University of KwaZulu-Natal*, Durban PhD thesis: 3 157
- Rathilal, S., Carsky, M., Heyberger, A., Rouskova, M. 2011. Correlations for the prediction of NTU and mass transfer coefficient for a VPE. *South African Journal of Chemical Engineering*. 16(1): 30-49.
- Saien. J., Daliri, S. 2008. Mass transfer coefficient in liquid-liquid extraction and the influence of aqueous phase pH. *Industrial and Engineering Chemistry Research*. 47(1): 171–175.
- Seader, J.D. Henley, E.J. 2006. Separation process principles, 2<sup>nd</sup> Edition, John Wiley & Sons, Inc., United States of America, Chapters 4 and 8.
- Shen. Z.J., Rama Rao, N.V., Baird, M.H.I. 1985. Mass transfer in a reciprocating plate extraction column – effect of mass transfer direction and plate material. *The Canadian Journal of Chemical Engineering*. 63(1): 29–36.
- Taylor, P.A., Baird, M.H.I., Kusuma, I. 1982. Computer control of holdup in a reciprocating plate extraction column. *The Canadian Journal of Chemical Engineering*. 60: 556-565.