

Difference of hydrodynamics for a VPE with and without mass transfer and effect of agitation level on extent of mass transfer

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Abstract-Liquid-liquid extraction offers an alternative separation technique, other than distillation, that uses a solvent for the separation of liquid constituents (Pratt, 1983). The vibrating plate extractor (VPE) is a mechanically agitated column that offers many advantages to conventional extraction equipment. Besides the dimensions of the column (diameter and height) and plate design, the VPE has many variables that may be appropriately adjusted to achieve the desired results viz. amplitude and frequency of vibrations, tray spacing, individual flow rates and the final throughput. The first part of the paper investigates the difference in hydrodynamics of the column due to the mass transfer process showing that the hydrodynamics (dispersed phase holdup, droplet size and size distribution) cannot be investigated without mass transfer in order to predict the behaviour during mass transfer. The second part of this paper is devoted to the investigation of the effect of agitation level on the extent of mass transfer.

INTRODUCTION

Liquid-liquid extraction may be achieved by many physical equipment that use either gravitational or centrifugal forces in unagitated or mechanically agitated equipment (Robbins, 1996; Lo, 1996; Humphrey et al, 1997; Lo et al, 1992). The VPE is a mechanically agitated column that uses vibrating sieve trays to achieve smaller droplets of the dispersed phase and in so doing, increase the area available for mass transfer. This is similar to the Karr column except that it has small diameter holes on the plates for the dispersed phase and downcomers for the continuous phase which effectively uses lesser energy to achieve the same mass transfer performance (Lo et al, 1983; Ioannou et al, 1976).

Many researchers have investigated hydrodynamics of columns without mass transfer however the mass transfer process has an effect on the hydrodynamics of the column resulting in different holdups and droplet sizes being obtained during mass transfer and in the absence of mass transfer (Rama Rao et al, 1991; Aravamudan et al, 1999; Baird et al, 1973; Bell et al, 1969).

This paper initially investigates the difference in hydrodynamics of the column (in terms of dispersed phase holdup, average droplet size and droplet size distribution) during mass transfer and when mass transfer does not take place.

The VPE has many variables that may be adjusted in order to improve the efficiency of the column and the latter part of this paper shows the effect of agitation level (product of amplitude and frequency of the plate vibrations), tray spacing and solvent to feed (S/F) ratio on the extent of mass transfer.

EXPERIMENTAL

The toluene-acetone-water system is recommended by the European Federation of Chemical Engineering as a standard test system for liquid extraction (EFCE, 1985) and was therefore chosen for this investigation. Toluene

and water are immiscible liquids and was used to investigate the hydrodynamics without mass transfer. The gas chromatograph (GC) was used for the analysis of the different phases and the toluene-acetone-water system was shown to have high accuracy and repeatability during analysis using gas chromatography (Saien et al, 2006).

The details of the experimental setup may be found in another paper (Rathilal et al, 2011) but essentially consisted of a glass tower with an ID of 47.7 mm and an effective height of 4.76 m. The trays were stainless steel sieve plates equipped with downcomers (fig. 1).

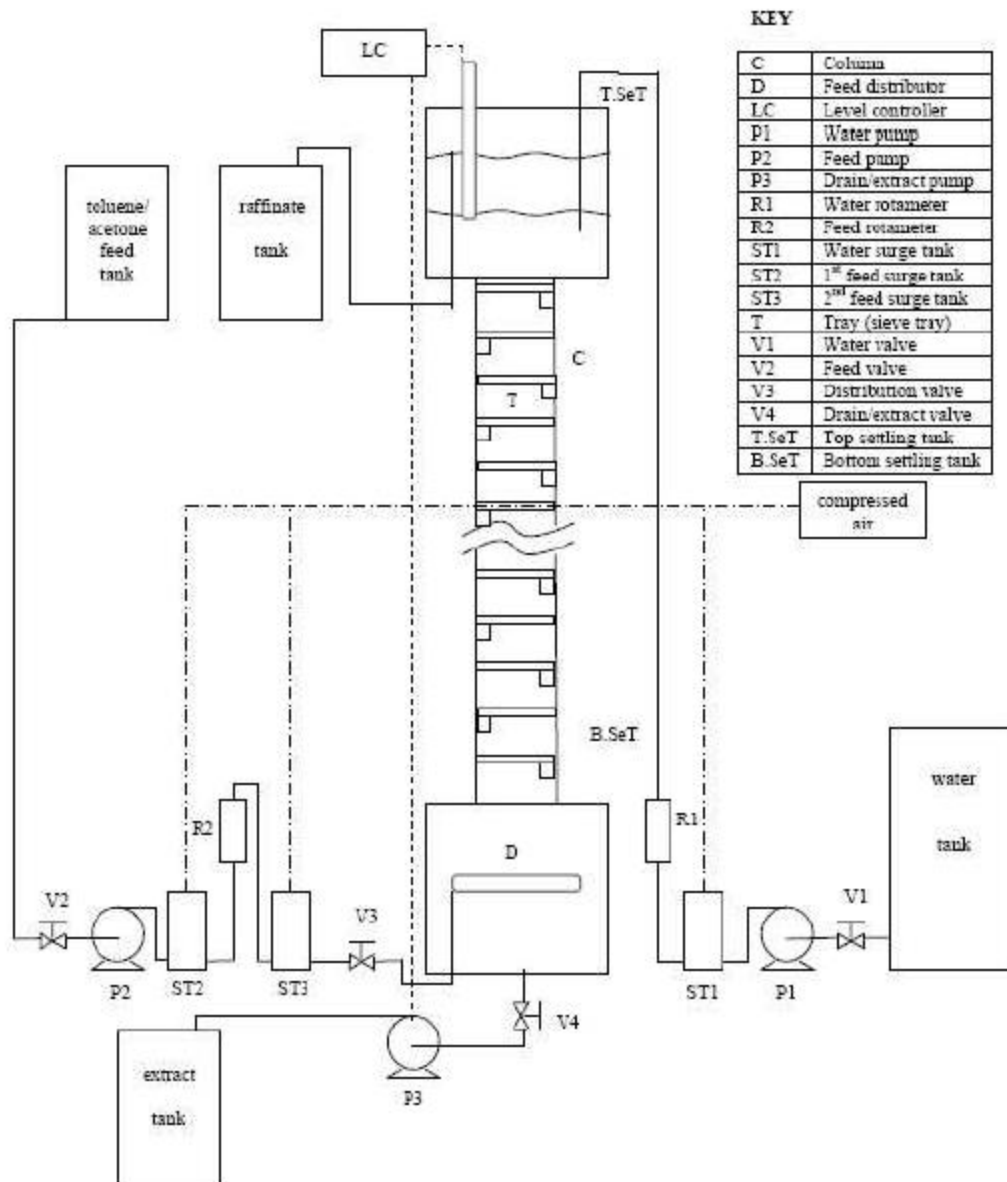


Fig. 1 – Experimental equipment and flow diagram (Rathilal et al, 2011)

The aqueous phase was used as the continuous phase and flowed down the column while the hydrocarbon phase (commercial grade toluene) was used as the dispersed phase flowing up the column due to the density difference between toluene and water (Laddha et al, 1983). During mass transfer acetone (commercial grade) was used as the solute and was transferred from the dispersed (feed mixture of 6% acetone in toluene) to the continuous phase. Therefore, the inputs to the column was essentially the feed (acetone + toluene) at the bottom of the column and the solvent (tap water) at the top of the column. The outputs were the extract at the bottom of the column (water + extracted acetone) and the raffinate at the top of the column (toluene + un-extracted acetone). A level controller was used to maintain the liquid-liquid interface in the upper settling tank.

The stainless steel plate stack, mounted on a central shaft, was connected eccentrically to a motor at the top of the column. The amplitude of the vibrations was set to 2.5 mm and the frequency was varied by adjusting the speed of the motor (Rathilal et al, 2011).

The surge tanks were designed to hold a maximum of 2 litres of liquid and were made of stainless steel. They were equipped with side transparent tubing so that the level of liquid could be seen. The purpose of the surge tanks was to reduce the fluctuations caused by the peristaltic motion of the pumps and the vibration of the trays in the column so that the flow rate may be read easily on a rotameter. The tanks were pressurized from the top with compressed air. The tank situated on the water line was between the pump and the rotameter since the only cause of the fluctuating flow rate was the peristaltic motion of the pump. This reduced the fluctuations drastically and allowed for a stable flow rate reading to be taken from the rotameter.

The organic line proved to be more problematic since the fluctuations were caused by two independent factors i.e. the peristaltic motion of the pump and the vibration of the plates in the column which caused a variation in pressure at the bottom of the tank where the dispersed phase entered. The surge system was designed such that one surge tank was placed between the pump and the rotameter to reduce the fluctuations caused by the pump while a second surge tank was placed between the rotameter and the distributor in the column which was used to reduce the fluctuations caused by the variation in pressure at the bottom of the column (as a result of the vibration of the plates). This allowed the operator to take a reading on the rotameter without large fluctuations in the rotameter reading. The reading did however change when the drain pump changed its speed between its upper and lower limits; however, this change was minimal. This allowed for very accurate flow measurements to be recorded.

The trays were designed in such a way that the free areas available for flow for the two phases were approximately the same. As a result, the solvent to feed (S/F) flow ratios were chosen to be around 1:1. Actual S/F ratios were 1:1, 1:2 and 2:1. The total throughput was kept constant at 30 l/h (Rathilal et al, 2011).

The design specifications of the sieve plates are given below:

Diameter = 47.4 mm

Thickness = 2 mm

Hole diameter = 2.98 mm (cylindrical holes)

No. of holes = 37

No. of plates = 47

Downcomer diameter = 10.9 mm/tube (3 tubes per tray)

Downcomer length = 43.3 mm

Cross sectional area of tray = $1.764 \times 10^{-3} \text{ m}^2$

Total area of holes = $0.258 \times 10^{-3} \text{ m}^2$

Free area for dispersed phase = 14.6 %

Total area of downcomers = $0.280 \times 10^{-3} \text{ m}^2$

Free area of continuous phase = 15.9%

The following equation was used to calculate the Sauter mean diameter to estimate the average drop diameter of the dispersed phase droplets (Baird et al, 1973).

$$d_{32} = \frac{6 \sum V_i}{\sum S_i} = \frac{6 \sum n_i d_i^3}{6 \sum n_i d_i^2} \quad (1)$$

When it was time to take measurements, the interface level was marked in the top settling tank. Samples were taken from the extract and raffinate phases and analysed on the GC. The water pump, feed pump and the distribution valve were all stopped / closed at the same time. It was not necessary to stop the raffinate flow as this stopped automatically since there was no input into the system.

The plates were vibrated at a frequency of around 1.5 Hz which corresponded to the lowest holdup frequency and the dispersed phase was allowed to accumulate in the top settling tank until all of the dispersed phase accumulated. When this process was complete the level of the toluene phase below the marked interface level was measured and the volume was calculated by multiplying this height by the area of the settling tank to give the holdup in the column.

The volume of liquid (water) occupied in the active part of the column was measured by collecting the water during a draining process starting at the top of the plate stack and ending at the bottom of the plate stack. This volume was measured as 7.8 litres. This is the volume that was occupied by the continuous phase when no dispersed phase was present (dispersed phase holdup = 0).

The measured volume of toluene accumulated was divided by 7.8 in order to calculate the volume fraction of the holdup. This value is represented as a percentage in the holdup graph (Aravamudan et al, 1999).

A Perspex box situated around a section of the column (between plates 14 and 15 when the plate spacing was 100 mm and between plates 7 and 8 from the bottom of the column when the plate spacing was 200 mm) was filled with the continuous phase (water) and was illuminated from the rear and the sides. This arrangement allowed high contrast photographs of the droplets to be taken while reducing the error on the size of the droplets caused by the curvature of the column (Rathilal et al, 2011). The camera used was a Panasonic Lumix DMC-FZ5 and the photographs were taken to incorporate the entire stage between two plates.

The photographs were analysed using an image analysis software (Image Tool) to evaluate the size of the droplets. At least 250 droplets were measured for each photograph (Rama Rao et al, 1991; Joseph et al, 1998; Lisa et al, 2003). The size distribution was calculated by expressing the number of droplets in 0.2 mm intervals as a percentage of the total number of droplets (Usman et al, 2006). This gave the percentage of droplets that occurred in a particular range.

All experimental work was carried out at ambient temperature. Each experimental run was repeated at least once. Three to five photographs were taken of the droplets during each run in order to obtain acceptable results of distribution and Sauter mean diameters. During the GC analysis, each sample was analysed at least 3 times with average values being taken as the final result. It was noticed that the results are highly repeatable.

RESULTS AND DISCUSSION

Time to reach stability

All readings for the experiments had to be taken when the system had reached a stable steady state (i.e. constant flow rates, constant holdup and droplet sizes and constant concentrations). In order to decide how long the experiments should run before stability was reached, preliminary runs were performed at the 3 different solvent to feed ratios of 1:1, 1:2 and 2:1 with no mass transfer while keeping the plate spacing at 100 mm. The holdup was measured after allowing the column to run for 20 minutes, then for 40 minutes, 60 minutes and finally for 80 minutes.

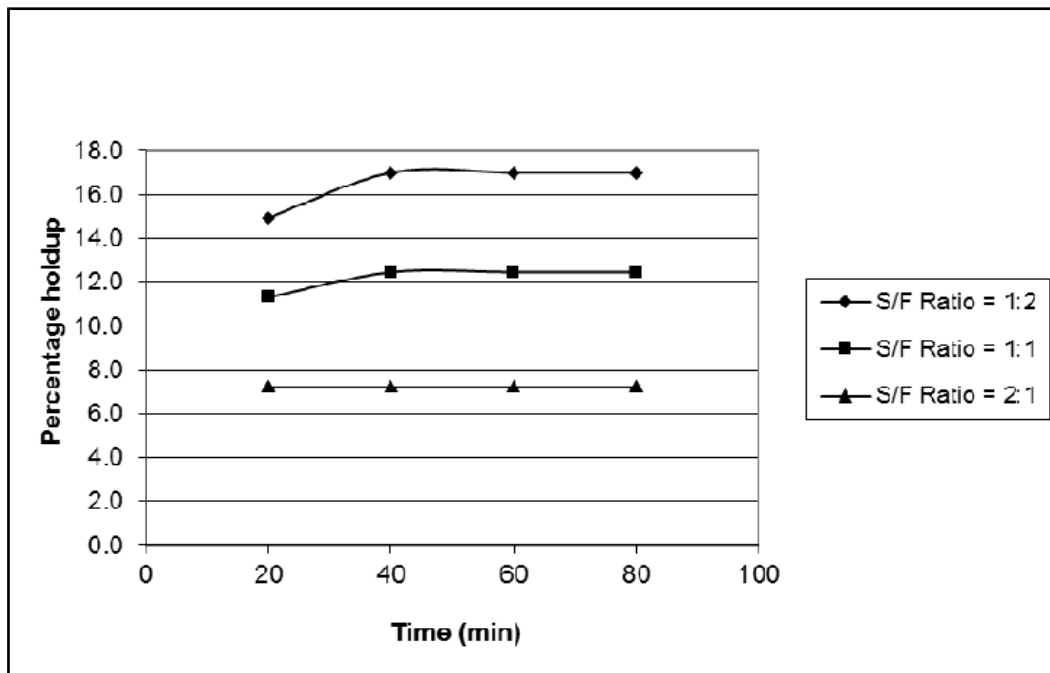
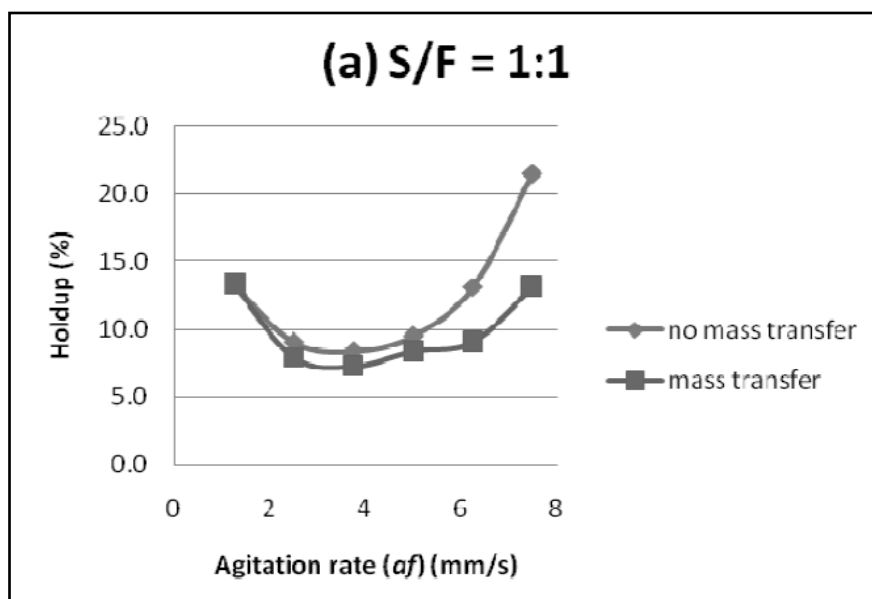


Fig. 2 – Time to reach stability

From fig. 2 it can be seen that 45 minutes is a sufficient time to allow the experiment to run before stability is reached in terms of holdup for all three flow ratios. It was assumed that this time is also sufficient for complete stability to be obtained in terms of droplet sizes and concentration profiles and as a result all the experiments were conducted for a minimum of 45 minutes. The assumption was justified by obtaining good repeatability for the balance of the experiments.

Holdup

Holdup was calculated for the different solvent to feed flow ratios and different agitation levels (*af*). The amplitude was kept constant at 2.5 mm and the frequency was increased from 0.5 Hz up until flooding occurred. As a result *af* increased from 1.25 mm/s (with increments of 1.25 mm/s) until flooding took place (Rathilal et al, 2011). The results are indicated in fig. 3 for the different flow ratios with and without mass transfer.



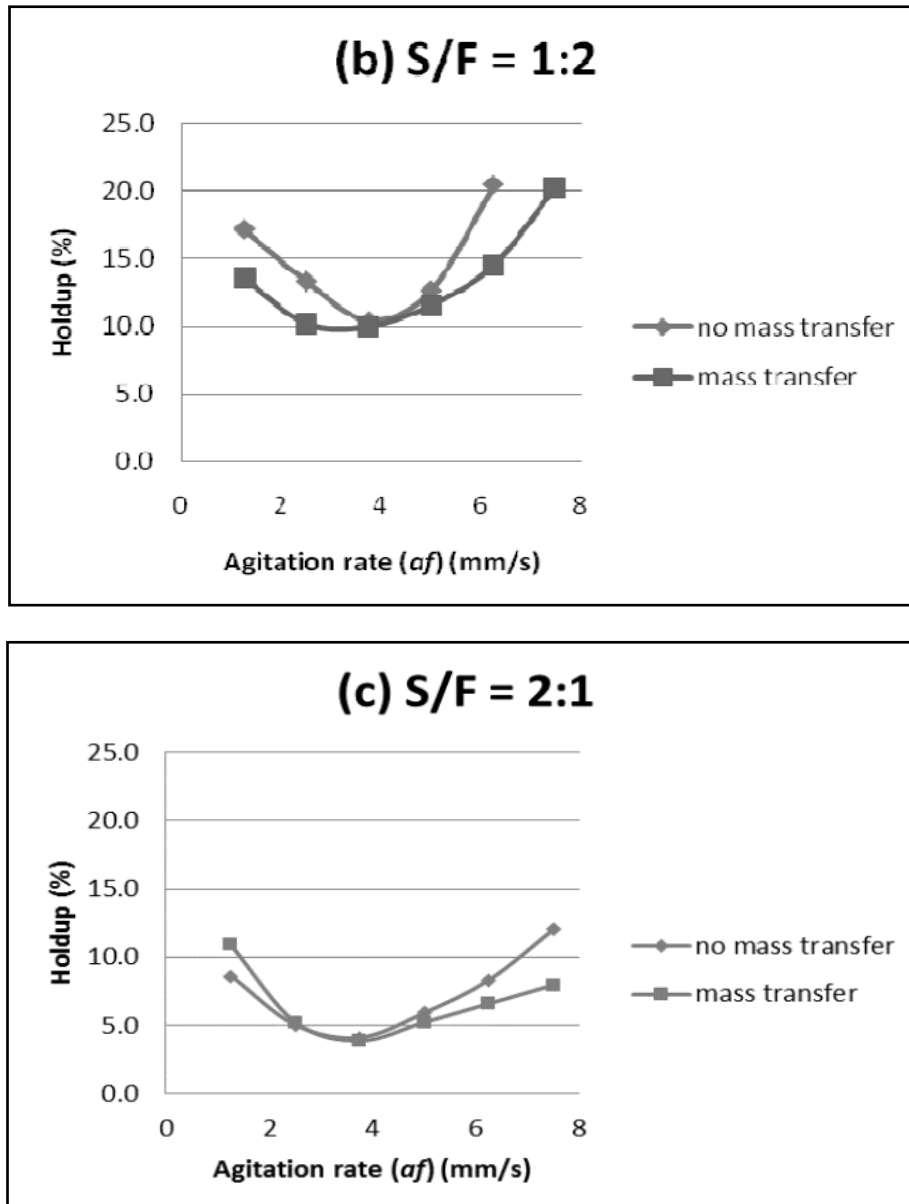


Fig. 3 – Effect of agitation level on holdup (tray spacing = 100 mm)

For all of the flow ratios investigated the holdup trends are similar; decreasing initially during the mixer-settler regime to a minimum at an agitation level of 3.75 mm/s and then starting to increase in the dispersion regime. Similar trends are observed in literature especially for the dispersed regime (Rathilal et al, 2011; Baird et al, 1973; Aravamudan et al, 1999; Baird et al, 1984). The holdup is initially high in the mixer settler regime due to a coalesced layer of dispersed phase being formed under the plates which decreases as the agitation level is increased. At higher agitation levels more, smaller droplets are formed increasing the holdup further. There is no evidence of a coalesced layer below the plates in the dispersion regime.

The transition from mixer-settler to dispersion regime (minimum holdup) is shown to be independent of flow rates and whether or not mass transfer takes place as it occurs at the same agitation level of 3.75 mm/s for all of the flow ratios tested.

It can, however, be seen that there is a difference between the holdup calculated with mass transfer compared to that without mass transfer with the former usually being lower. This is due to the fact that the solute (acetone) is continuously being removed from the dispersed phase and as a result there is lesser dispersed phase during mass transfer than in the absence of mass transfer which results in the lower holdup. As a result, the column may be

run at higher agitation levels with mass transfer than in the absence of mass transfer before flooding occurs confirming the observations made by Shen et al (1985). This also proves that predictions of mass transfer may not be done using measurements of holdup in the absence of mass transfer as the measurements are affected by mass transfer. These results were for a relatively low feed concentration (6%) and the differences in holdup may be much larger for higher concentrations.

Droplet size distribution

Photographs of the droplets (through the Perspex box) were taken for each run and the size distribution of the droplets was determined by analysing these photographs. The results for the S/F ratio of 1:1 with and without mass transfer at low and high frequencies are shown in fig. 4. The other flow ratios showed similar trends.

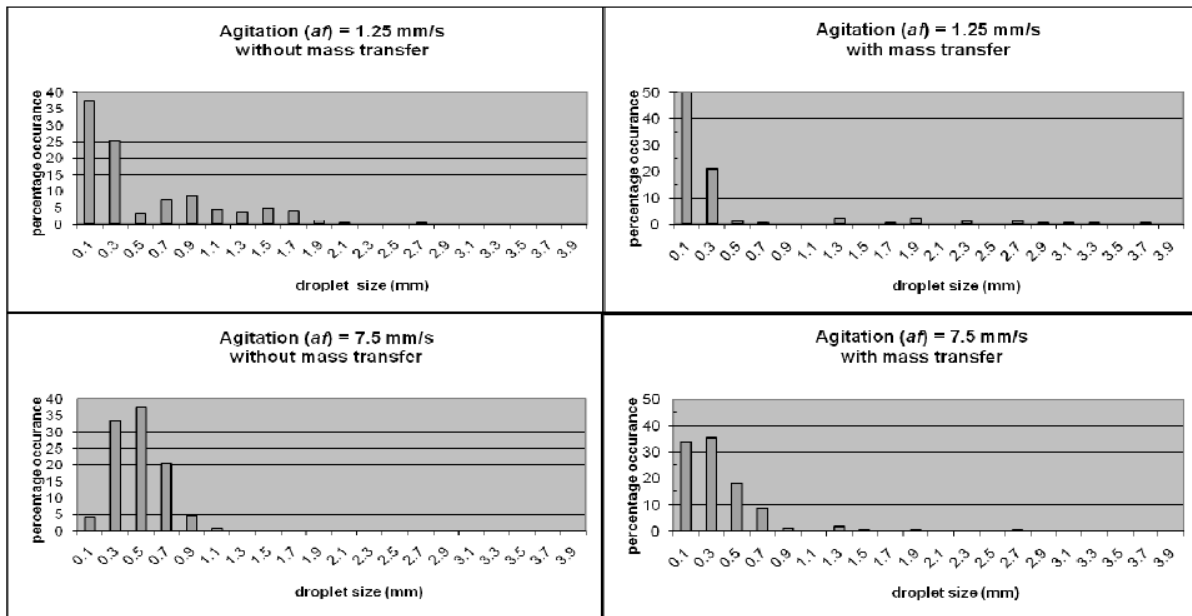


Fig. 4 – Droplet size distribution for S/F = 1:1 (tray spacing = 100 mm)

Fig. 4 indicates that there is a wide variation of droplet sizes at low agitation levels, while higher agitation levels create a more uniform size distribution having diameters that are much smaller. Although only four size distributions are shown in this paper, the distributions for other flow ratios and agitation levels showed the same trends. These size distributions together with equation (1) were used to calculate the Sauter mean diameter (Rathilal et al, 2011).

During mass transfer, there is still some large droplets present even at high agitation levels due to the enhanced coalescence effect during mass transfer. As two drops approach each other, the surface tension is reduced due to the solute moving from the dispersed phase to the continuous phase. This causes the continuous phase between the drops to be drained and the drops coalesce forming bigger drops. This is consistent with literature (Aravamudan et al, 1999; Shen et al, 1985). Once again, this also proves that predictions of mass transfer may not be done using measurements of drop size distribution in the absence of mass transfer as the measurements are affected by mass transfer.

Sauter mean diameter (d_{32})

The results for the three different flow ratios at varying agitation levels with and without mass transfer are shown in fig. 5.

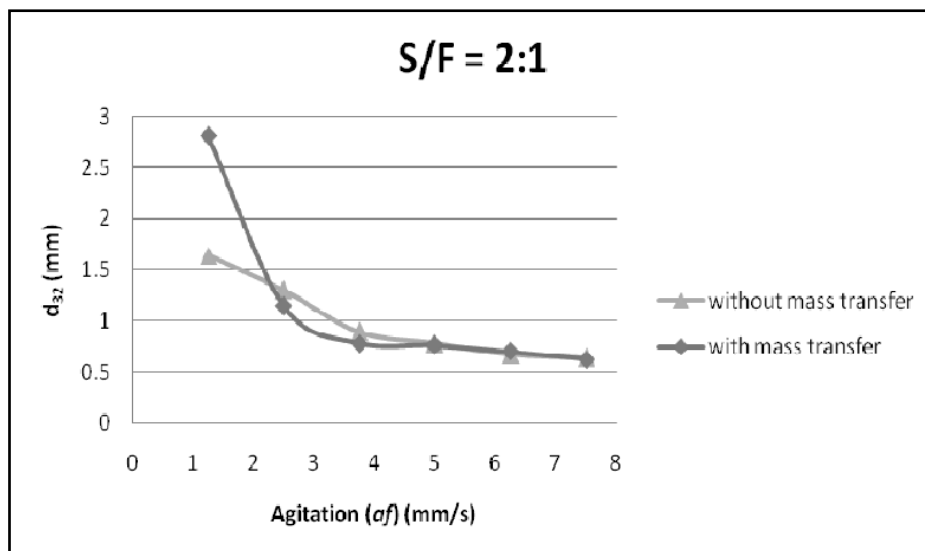
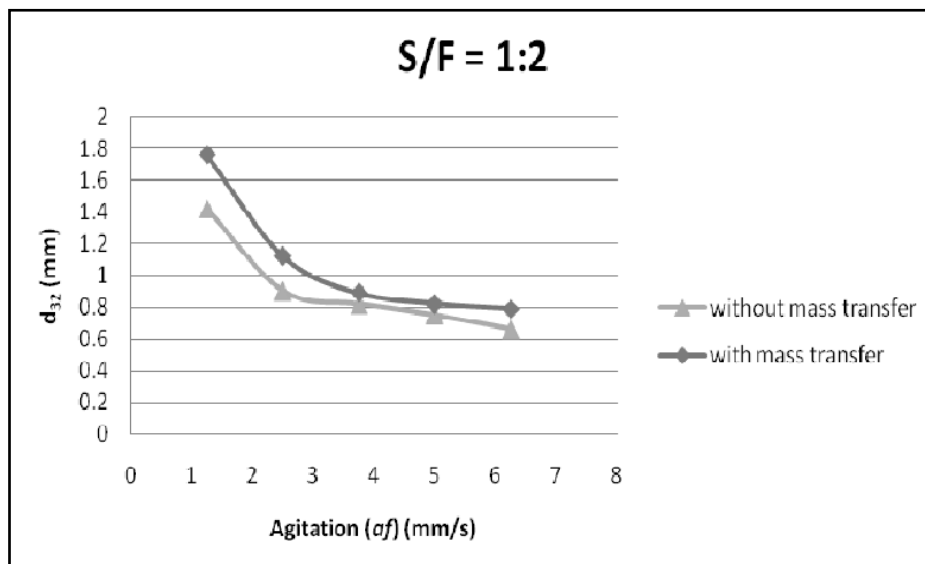
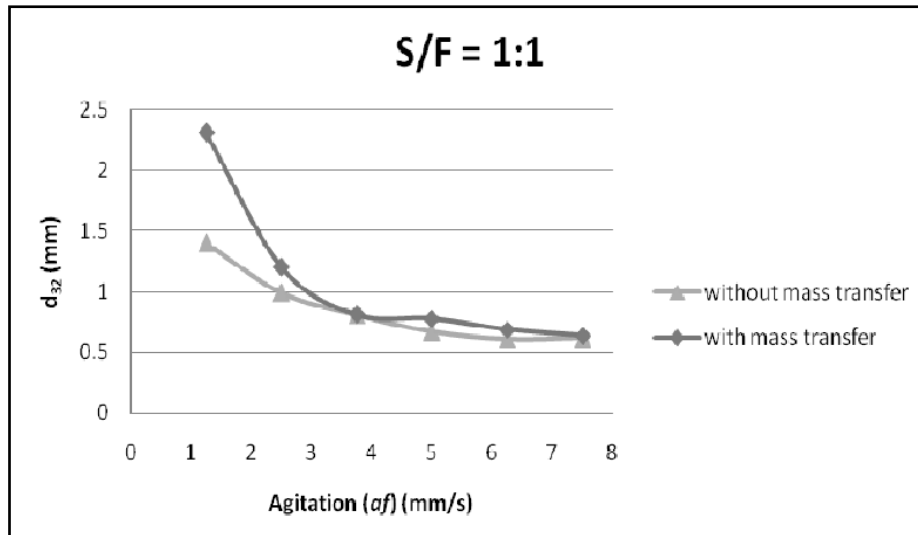


Fig. 5 – Sauter Mean Diameter (tray spacing = 100 mm)

As the agitation level is increased, smaller droplets are formed and this is shown in fig. 5 where the Sauter mean diameter is seen to decrease. In the mixer-settler regime there seems to be a significant difference in the Sauter mean diameter during mass transfer and in the absence of mass transfer. During the dispersion regime, the difference in diameter is, however, minimal (Rathilal et al, 2011). The diameters are generally larger for the case when mass transfer occurs. This is due to the enhanced coalescence effect during mass transfer thus forming larger droplets. This provides more proof that predictions of mass transfer may not be done using measurements of Sauter mean diameter in the absence of mass transfer as the measurements are affected by mass transfer.

Extent of Mass Transfer

The extent of mass transfer was measured by analysing the extract and raffinate phases to determine their concentrations (using a GC). The effect of agitation level on the extent of mass transfer for the three flow ratios and different plate spacing is illustrated in fig. 6. In addition to varying the S/F ratios, the tray spacing was also increased for the 1:1 S/F ratio.

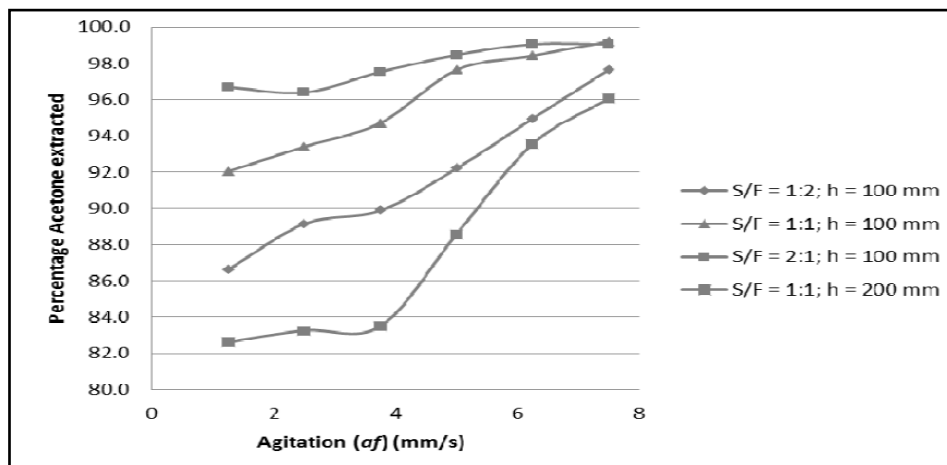


Fig. 6 – Effect of agitation level and plate spacing on the extent of extraction

The percentage acetone extracted is calculated by subtracting the raffinate concentration from the feed concentration and expressing this difference as a percentage of the original feed concentration. The effect of increasing agitation level is clearly indicated in fig. 6 showing that the amount of acetone extracted increases as the agitation level is increased with higher values being obtained when the S/F ratio is increased. From the holdup chart we have seen that an increase in agitation level increases the holdup and the d_{32} chart shows that this is accompanied by a larger number of droplets with smaller mean diameters. The total effect of this is that the interfacial area available for mass transfer is increased and therefore the effectiveness of the extraction should be improved. As shown in fig. 6, this corresponds to an increased amount of acetone being extracted.

The reason that the effectiveness of extraction is higher for higher S/F ratios is because there is more solvent available to remove the acetone and the concentration gradient is increased. This in effect will improve the extraction effectiveness.

When the plate spacing is increased (to 200 mm), the extraction effectiveness is drastically reduced due to a fewer number of transfer units being present for the mass transfer to take place. However, the column was not near its flooding condition at an agitation level of 7.5 mm/s like it was during the 100 mm tray spacing. As a result the agitation level may be increased beyond this limit before flooding can take place resulting in a greater extraction effectiveness being achieved.

CONCLUSION

Predictions of mass transfer may not be made by analysing the hydrodynamics of a VPE without mass transfer taking place. The holdup during mass transfer is substantially lower than when no mass transfer occurs due to

solute being transferred from the dispersed phase to the continuous phase. The reverse may be true for the transfer of solute from the continuous phase to the dispersed phase where the holdup will be higher than when no mass transfer occurs. The size distribution is wider during mass transfer and the Sauter mean diameter is larger (especially in the mixer settler regime) due to the enhanced coalescence effect during mass transfer. Thus for the measurement of hydrodynamics of this mass transfer equipment (VPE), mass transfer must take place.

The VPE has many variables that may be adjusted independently or as a group in order to improve the efficiency of mass transfer. The main variable is the agitation level (product of amplitude and frequency of plate vibration). As agitation level is increased, the extent of mass transfer may be increased substantially. The column may be operated at a stable steady state very close to flooding conditions.

NOMENCLATURE

a = amplitude (half-stroke), mm

af = agitation rate (mm/s)

d_i = dispersed phase drop diameter, m

d_{32} = Sauter mean drop diameter, m

f = frequency, Hz

h = centre to centre plate spacing, mm

n_i = number of drops

S/F = solvent to feed ratio

S_i = surface area of a drop, m^2

V_i = volume of a drop, m^3

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