Correlations for the prediction of NTU and mass transfer coefficient for a VPE

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Abstract

Liquid-liquid extraction is the separation of the constituents of a liquid solution by contact with another insoluble. The reciprocating column (RPC) and vibrating plate column (VPE) are two types of mechanically aided columns. This paper aims at developing appropriate correlations for the prediction of *NTU/HETS* and the mass transfer coefficient, k_{ox} for the VPE based on the agitation level of the plates (*af* – the product of frequency and amplitude of the plate motion), the plate spacing and the flow rates which will allow for the simplification in the design of this type of column. The system chosen was the acetone-toluene-water system (standard test system – EFCE) with the acetone in toluene forming the feed that is dispersed in the column as it moved upward while the water moved as a continuous phase down the column. Experiments were conducted to evaluate the hydrodynamics of the droplets moving up the column and to evaluate the mass transfer that occurs (in order to evaluate *NTU, HTU* and *kox*) while varying the agitation levels and spacing of the plates in the column. Successful correlations were developed using some of the experimental data and these correlations were verified with additional data.

1. Introduction

Many devices are available for liquid extraction that may use the gravitational force or centrifugal force for the separation and may be unagitated or mechanically agitated in order to create finer dispersions and improve the mass transfer [3][4][5][6]. The reciprocating column (RPC) and vibrating plate column (VPE) are 2 types of mechanically aided columns. A lot of research has gone into the development of mechanically aided extraction columns but is limited when it comes to RPC and VPE and most of this research is devoted to the RPC [7][8]. The essential difference between these two types of columns is that the VPE has trays with smaller

diameter holes (resulting in a smaller free area) and downcomers whereas the RPC (Karr column) has a larger free area with no downcomers. The VPE column operates at relatively low amplitudes and frequencies (compared to the RPC) which implies low mechanical stress and energy consumption [9]. The RPC uses more energy to achieve the same mass transfer performance than the VPE [10].

The dispersion regimes that may exist in a typical VPE are the mixer-settler regime, the dispersion regime and the emulsion regime as described by [11] and [12]. The RPC may only operate in the emulsion regime while the VPE may be operated in the mixer-settler regime as well as the emulsion regime [7][9][13].

The purpose of this research was to investigate the effects of the various variables and develop correlations in order to optimise the performance of the VPE. It was proposed to formulate correlations for the prediction of the number of transfer units (taking into account axial dispersion) as well as the mass transfer coefficient as functions of the variables stated above.

As a result, this paper shows investigations of the effects of agitation level (product of amplitude and frequency of the plate vibrations), S/F ratio and tray spacing on the extent of mass transfer, *NTU* and the mass transfer coefficient by investigating variables such as drop size/distribution and holdup. Appropriate correlations for the prediction of *NTU* and mass transfer coefficient are developed empirically and the correlations are tested with additional data.

2. Experimental

The toluene-acetone-water system was chosen as it is the standard system recommended by the European Federation of Chemical Engineering as a test system for liquid extraction [2]. Toluene and water are considered to be completely immiscible in each other. The toluene-acetone-water system is also preferred as it has high accuracy and repeatability when using gas chromatography for the analysis [14].

Water was used as the continuous phase flowing down the column while toluene was used as the dispersed phase flowing up the column (fig. 1) due to the density difference between toluene and water [15]. As a result of this arrangement an interface between the two immiscible phases (water and toluene) was located at the top of the column in the upper settling tank [5].

Mass transfer took place from the dispersed (feed mixture of 6% acetone in toluene) to the continuous phase.
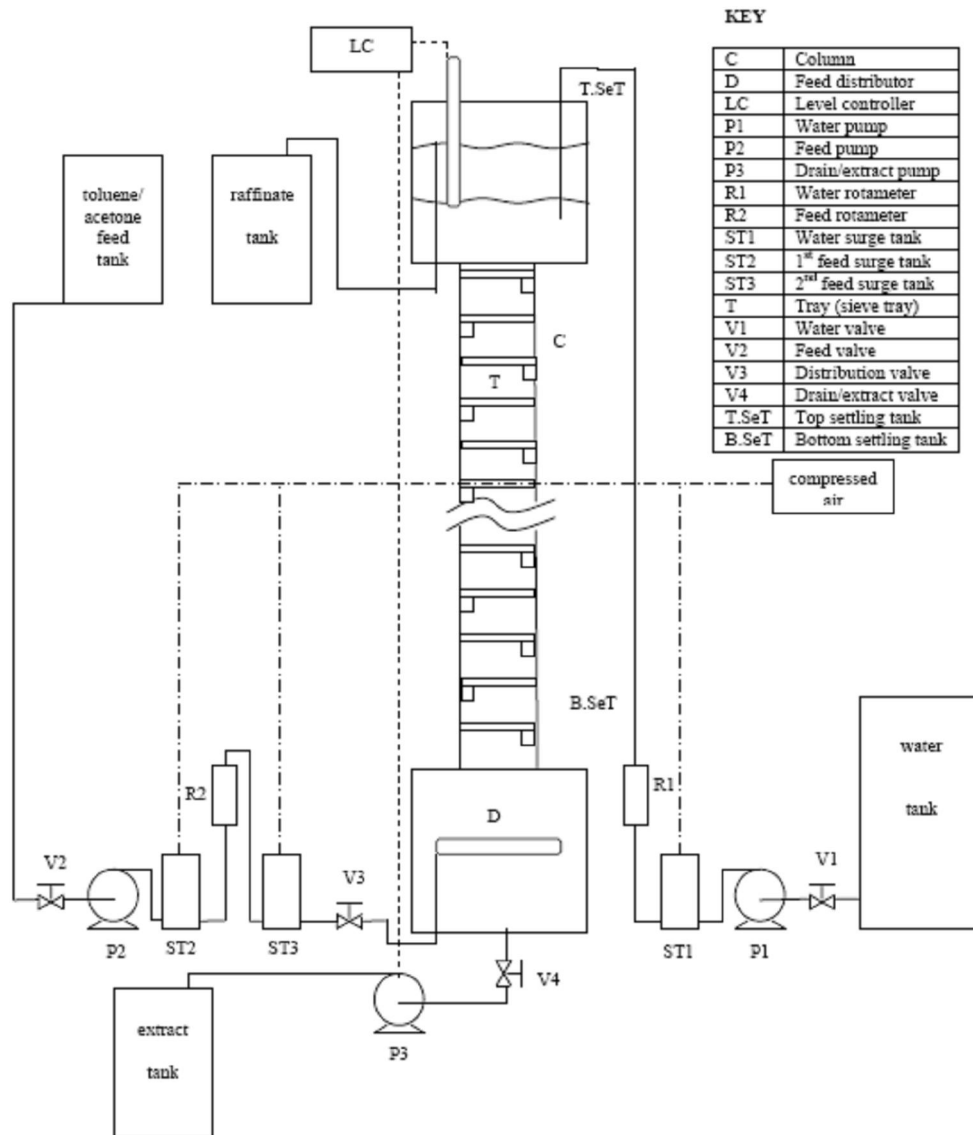

Fig. 1 – Experimental equipment and flow diagram

The active part of the column was made up of 8 flanged glass cylinders (ID = 47.7 mm) forming an effective height of 4.76 m. The upper and lower settling tanks where broader in diameter having an ID of 150 mm. The trays were stainless steel sieve plates with 37 holes (diameter = 3 mm) and 3 cylindrical downcomers (diameter = 11 mm). The small holes on the plate allowed for the dispersed phase to flow up the column and to be re-dispersed after each plate. The downcomers were arranged so that consecutive plates had the downcomers on opposite sides. This allowed for the continuous phase to flow across the plate as it moved down

the column. The trays were made of stainless steel which is preferentially wetted by water (as opposed to toluene) and as a result allowed the water to be the continuous phase.

A level controller consisting of a conductivity probe, a control box that housed the controller electronics and a variable speed pump, which was the same pump used for the drain/extract, was used to control the liquid-liquid interface in the top settling tank due to the difference in electrical conductivity of water and the hydrocarbon fluid (toluene). This information was used to control the speed of the drain pump and as a result kept the interface level constant. All the pumps were positive displacement pumps that used a rotor to control the amount of liquid that passed through silicon tubing. They were all variable speed pumps and the flow rates were adjusted by changing the speed of the pump.

A Perspex box was designed and placed on the outside of the column between plates 14 and 15 (when the plate spacing was 100 mm) and between plates 7 and 8 (when the plate spacing was 200 mm) from the bottom of the column in order to reduce the error on the size of the droplets caused by the curvature of the column during the photography of the drops. The box was illuminated from the rear and the sides to allow a high contrast photo to be taken.

The plate stack was mounted on a central shaft which was connected eccentrically to a motor at the top of the column. By adjusting the spacing between the connection point and the centre of the motor through an adjustable yoke, the amplitude of the vibrations could be changed. By adjusting the speed of the motor, the frequency of the vibrations could be varied.

Four pairs of samplers were placed along the length of the column in order to extract each of the phases independently from different positions in the column and evaluate the concentration at that particular position in the column. The continuous (aqueous) phase sampler had a stainless-steel tip and was positioned to face upwards (continuous phase flows down the column). The wetting preference of steel is the aqueous phase (as opposed to the organic phase) and it was possible to remove just the continuous phase from that sampler. The dispersed (organic) phase sampler had a Teflon tip and was faced downwards opposite to the flow direction of the dispersed phase. Teflon has a preferred wettability for the organic phase and as a result it was possible to remove only the organic phase from that sampler.

Since the free area available for flow for the dispersed and continuous phases was approximately the same (14.6 and 15.9 %, respectively) the solvent to feed (S/F) flow ratios were chosen to be around 1:1. Experiments were performed for three S/F ratios (1:1, 1:2 and

2:1) while keeping the total throughput (combined flow rates of dispersed and continuous phases) the same (at 30 l/h).

The Sauter mean diameter was used as an estimation of the average drop diameter of the dispersed phase in the column or section of the column. The following equation was used to calculate the Sauter mean diameter from experimental values [17].

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

When the system had reached steady state, samples were taken from the eight samplers along the length of the column as well as from the extract and raffinate tubes. These samples were analysed on a GC to evaluate the concentrations.

3. Results and Discussion

Holdup

Holdup was calculated for 3 different solvent to feed flow ratios of 1:1, 1:2 and 2:1 for agitation levels (*af*) starting at 1.25 mm/s increasing by 1.25 mm/s up until flooding occurred in the column (NB. The amplitude of 2.5 mm was kept constant and the frequency was increased from 0.5 Hz at 0.5 Hz intervals up until flooding occurred). The total throughput was kept constant at 30 l/hr and the feed flow rate was fixed at 10, 15 and 20 l/hr for the three different flow ratios. The results are indicated in fig. 2.
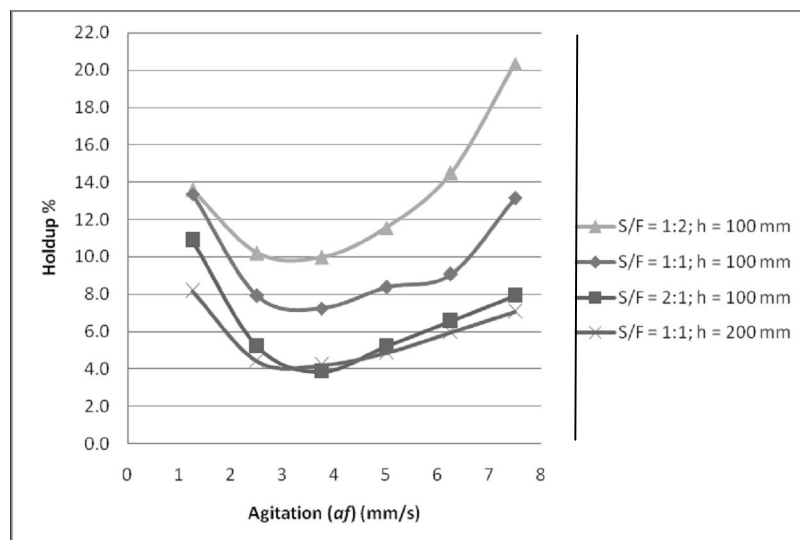

Fig. 2 – Effect of agitation on holdup

The holdup decreases initially during the mixer-settler regime to a minimum at an agitation level of 3.75 mm/s and then starts to increase in the dispersion regime. Initially the holdup is high due to the fact that the system is operating in the mixer-settler regime where most of the holdup is due to a layer of the dispersed phase (coalesced) being maintained under each tray. As the agitation level is increased, this layer is reduced and as a result the holdup is reduced. The holdup eventually reaches a minimum value which corresponds to the transition from mixer-settler to dispersion regime. As frequency is increased from this point, the holdup increases because the vibrating plates cause the droplet sizes to be decreased and more droplets are formed. Some small droplets are seen to circulate in a particular stage instead of moving vertically upwards all of the time which increases the residence time of the droplets in the stage (and the column) and thus also increases the holdup for a given flow ratio. As the agitation is increased further the holdup is expected to increase exponentially as the system moves towards the emulsion regime and then becomes unstable as the column approaches the flooding condition.

It is also noted that the holdup when the plate spacing was changed to 200 mm is much lower than when the plate spacing was 100 mm. In the mixer-settler regime there is a large noticeable layer of dispersed phase under the plates while during the dispersion regime there is a very slight layer of dispersed phase under the plates. When the plate spacing was increased the dispersed phase did not have time to accumulate under the plates and as a result the layer that formed under the plates was minimal. The transition from mixer-settler to dispersion regime (minimum holdup) is shown to be independent of flow rates as it occurs at the same agitation level of 3.75 mm/s for all of the flow ratios tested.

The holdup trends are consistent with literature. Most of the literature, however, showed trends mostly for the Karr column (which does not operate in the mixer-settler regime) and as a result only the second half of the trend was shown where the holdup increased exponentially from a minimum holdup [16][17][18].

At any particular agitation level the holdup for the S/F ratio of 1:2 is always higher than that for the flow ratio of 1:1 which in turn is always higher than that for the ratio of 2:1 indicating that the holdup decreases as the S/F flow ratio increases. As the toluene flow is decreased there is less dispersed phase in the column and as such the holdup decreases.

Experiments could not be carried out at an agitation level of 7.5 mm/s for the flow ratio of 1:2 or for higher agitation levels for the other ratios as flooding was seen to occur in the column.

Holdup is expected to increase unstably as flooding is approached and eventually when flooding occurs.

Droplet size distribution

At each agitation level for the different flow ratios photos were taken of the droplets and analysed to determine the size distribution of the droplets. The results for the S/F ratio of 1:1 with h = 100 mm are shown in fig. 3. The other flow ratios showed similar trends.

It is clear from the graphs that there is a greater size distribution at lower agitation levels and much smaller distribution at higher agitation levels indicating that as agitation level is increased the droplets move from being of a wide variation of sizes to being more uniform (and smaller) in size. The same observation was observed with the other flow ratios as well as when the plate spacing was changed to 200 mm. It is this size distribution that is used to calculate the Sauter mean diameter. Also noticeable from the graphs is that at low agitation levels a multi-modal distribution is visible while at higher agitation levels a uni-modal distribution is seen. This confirms the observation by [19] that under low agitation levels a dual mechanism of drop breakup exists (i.e. the flow through the perforations and the collision with the plates) while at higher agitation levels, the collision with the plates predominates, forming a uni-modal distribution.

Sauter mean diameter

The Sauter mean diameter was calculated from the size distribution using equation 1. The results for the three different flow ratios at varying agitation levels and tray spacing are shown in fig. 4.

The Sauter mean diameter decreases as the agitation level is increased due to more, smaller droplets being formed. There is a difference between the diameters for the different flow ratios in the mixer-settler regime; however the differences are minimal in the dispersion regime.

The Sauter mean diameter for the increased tray spacing case is shown to be much higher. Once again this is due to the reduced coalescence and breakup of the dispersed phase droplets because of the reduced number of plates since most of the coalescence and breakup occurs in the vicinity of the plates. The fewer number of plates in the column also reduces the amount of energy dissipated to the fluids which result in the drops not being as small as when there were more plates in the column dissipating more energy to the fluids.
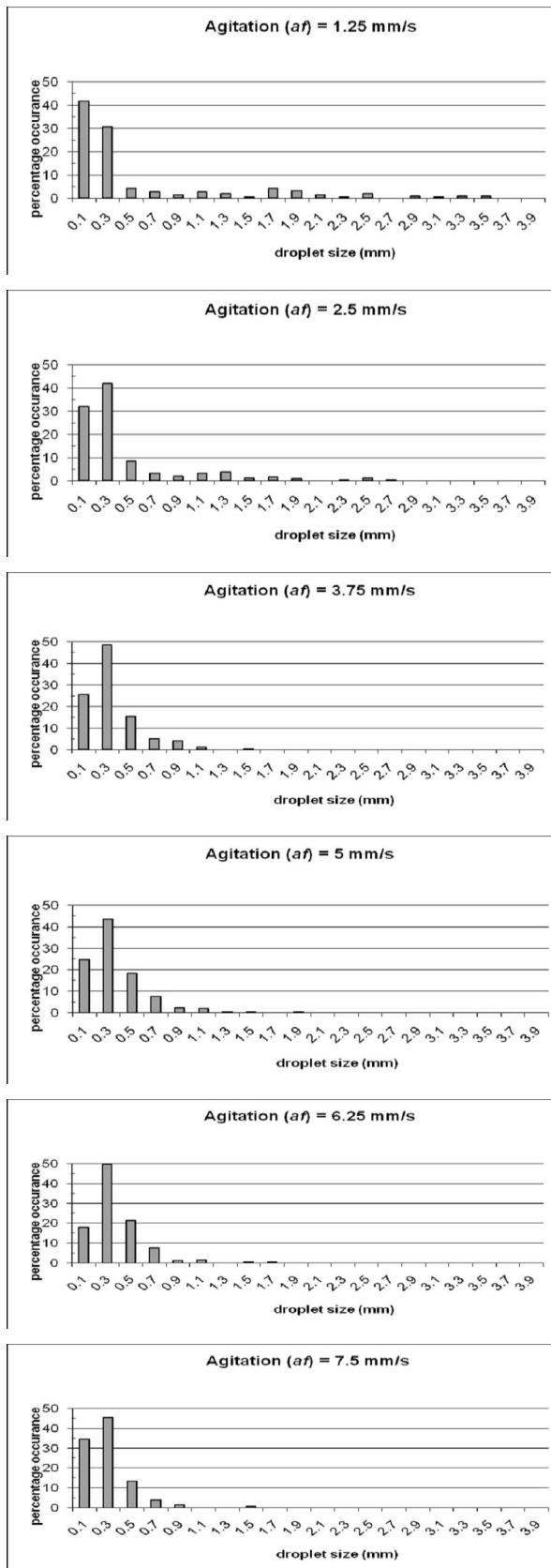
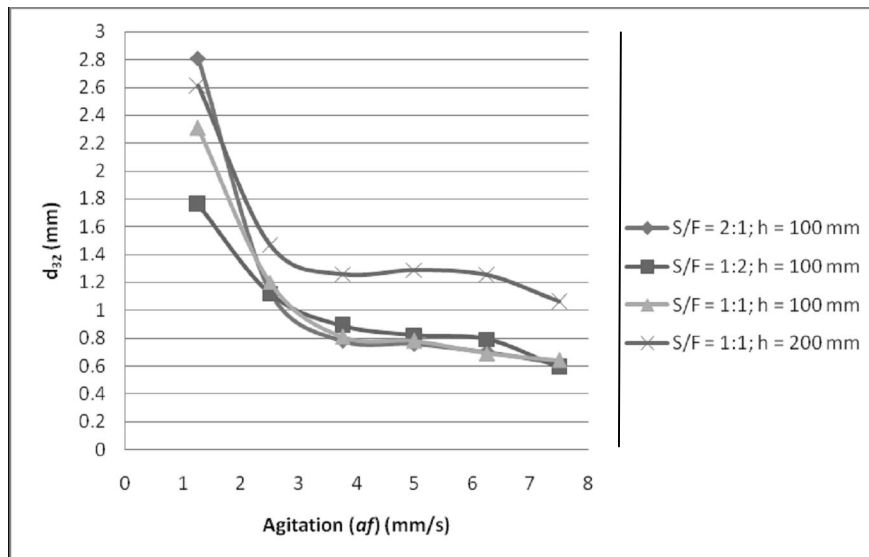Fig. 3 – Droplet size distribution for S/F = 1:1; h = 100 mm

Fig. 4 – Sauter Mean Diameter

Calculation of NTU

The true number of transfer units (N_{ox}) was obtained from the experimental equipment and was defined as the section between 2 consecutive plates. As a result for the tray spacing of 100 mm, there were 47 plates present corresponding to 46 true number of transfer units while for the tray spacing of 200 mm, 24 plates were present resulting in 23 true number of transfer units.

In order to find the ideal/theoretical number of transfer units, concentrations of both phases were evaluated from taking samples along the length of the column. This enabled the operating line to be obtained. The equilibrium line for the acetone-toluene-water system is linear with a slope of 0.832 [14][20]. Fig. 5 shows the graph for a solvent to feed ratio of 1:1, a tray spacing of 100 mm and an agitation level of 1.25 mm/s. It can be seen from the operating line (fairly linear) that the fluids essentially move in a plug flow manner (indicated by a straight operating line) with minimal backmixing in the dispersed phase.
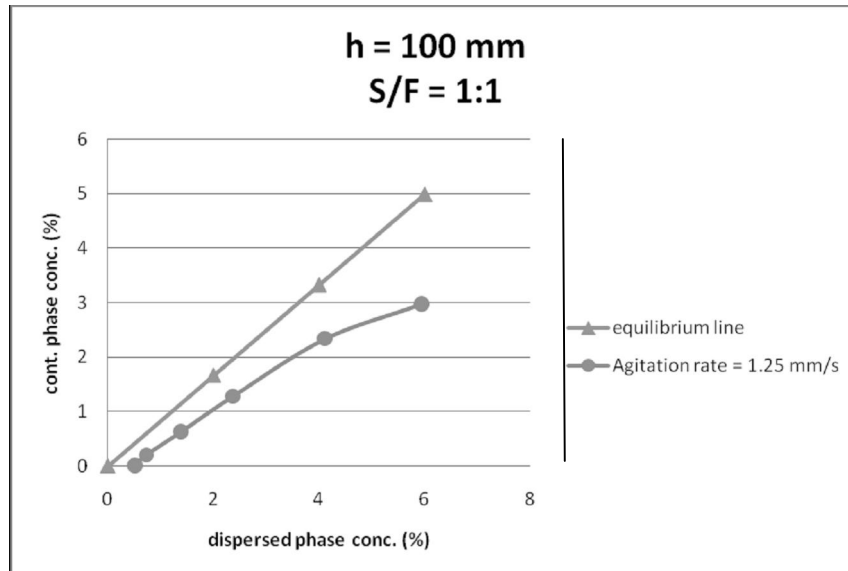
Fig. 5 – Operating line construction (minimum agitation rate)

The ideal measured *NTU* (N_{oxm}) may be obtained by stepping off between the operating line and equilibrium line on the *y* versus *x* curve similar to the McCabe Thiele method in distillation [3]. When the operating line is straight (indicating no backmixing), the ideal *NTU* (plug flow) may be calculated from the following equation [21]:

$$N_{oxp} = \frac{1}{[(y_i - y_o)/m(x_o - x_i)] - 1} \ln \frac{(y_i - mx_o)}{(y_o - mx_i)} \qquad (2)$$

where N_{oxp} is the plug flow overall *NTU* based on the *x* phase and the subscripts *i* and *o* refer to the inlet and outlet concentrations, respectively of the *x* and *y* phases.

Backmixing results in the operating line having a phase shift towards the equilibrium line resulting in a higher value for *NTU*, therefore:

$$N_{oxp} \leq N_{oxm}$$
$$(3)$$

As agitation level is increased, the amount of backmixing in the dispersed phase also increases. This is shown in fig. 6 for a solvent to feed ratio of 1:1, a tray spacing of 100 mm and an agitation level of 7.5 mm/s.
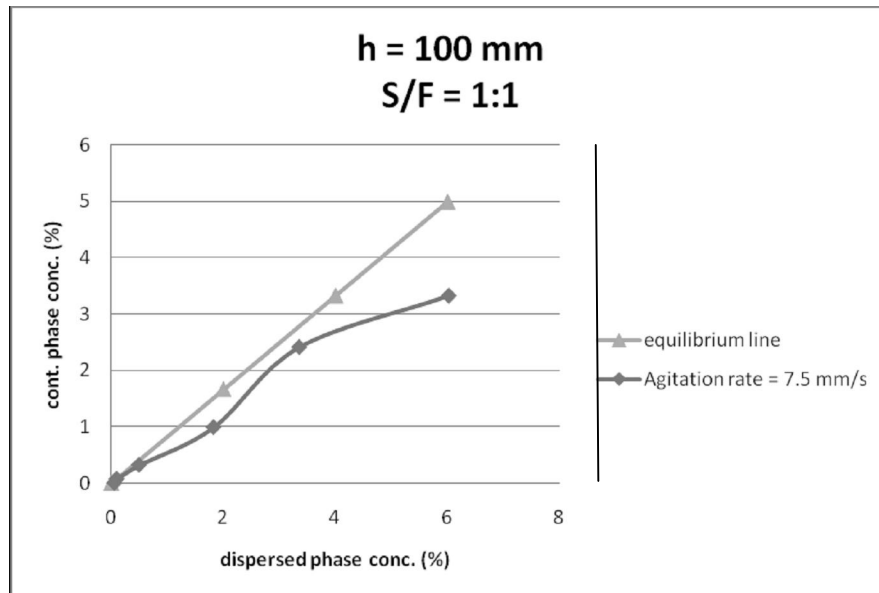
Fig. 6 – Operating line construction (maximum agitation rate)

It is clear from this graph that $N_{oxm} > N_{oxp}$ due to the fact that at this agitation level there is evidence of backmixing in the dispersed phase. As agitation level is increased the layer of dispersed phase that exists under the plates is reduced. This layer, when present, prevents droplets of the dispersed phase from re-entering the previous stage and thus prevents backmixing in this phase. For the S/F ratio = 1:2, there was significant backmixing for all agitation levels due to there being a large number of dispersed phase droplets being present causing re-circulation in the stage and resulting in some of the dispersed phase re-entering the previous stage. For the S/F ratio = 2:1 case, there was essentially no backmixing because there was always a layer of dispersed phase under the plates that prevented droplets from re-entering the previous stage.

All the *NTU* values were rounded off upwards so that if a fraction of a transfer unit was found the *NTU* was increased to the next whole number. Table 1 summarises the calculations for N_{oxm} and includes the values for N_{oxp} for comparison.

Table 1. Comparison of N_{oxm} and N_{oxp}

plate spacing	$h = 100$ mm						$h = 200$ mm	
solvent/feed ratio	S/F = 1:2		S/F = 1:1		S/F = 2:1		S/F = 1:1	
Agitation level (mm/s)	N_{oxm}	N_{oxp}	N_{oxm}	N_{oxp}	N_{oxm}	N_{oxp}	N_{oxm}	N_{oxp}
1.25	12	5	5	5	4	5	3	3
2.5	12	6	5	5	4	5	3	3
3.75	12	7	6	6	4	5	3	3
5	14	8	7	8	5	6	4	4
6.25	19	11	10	10	5	7	5	5
7.5	26	13	15	12	5	7	6	6

As can be seen for most cases $N_{oxp} \leq N_{oxm}$. There are some discrepancies especially for the S/F ratio of 2:1. This could be attributed to some experimental error in obtaining the samples along the length of the column. Another possible scenario is the case of forward mixing where some of the dispersed phase droplets move up the downcomers of the tray above and effectively skip a stage and mixes with the next consecutive stage. Very fine droplets may also experience forward mixing where they bypass a certain stage and effectively have a zero residence time in that stage.

Development of a Model for the Prediction of NTU

As can be seen from the discussion above, some of the dependent variables for *NTU* are the agitation level (product of amplitude and frequency of vibration), the solvent to feed flow ratio and the plate spacing. Fig. 7 shows this dependence for the flow ratio of 1:1 for both tray spacings.
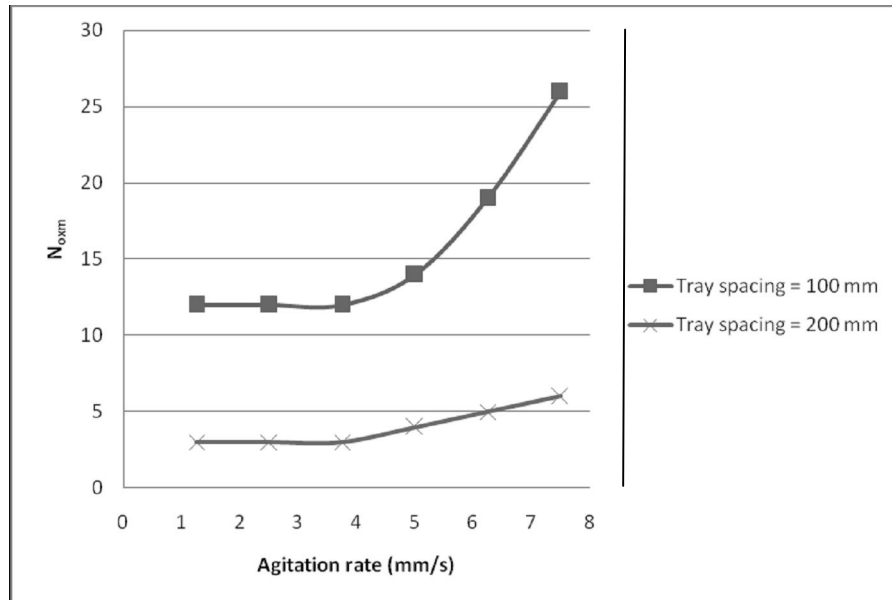
Fig. 7 – *NTU* correlation

NTU remains constant until an agitation rate of 3.75 mm/s (which corresponds to the minimum holdup when the transition occurs from mixer-settler to dispersion regime) and then starts to increase exponentially.

The NTU correlation has been developed that best describes the data above is given as follows:

$$N_{oxm} = \left[5.5 + \left(1.8e^{0.28(af)} - 5.5\right)u\right]L\left(\frac{100}{h}\right) \tag{4}$$

where L is the feed to solvent ratio (reciprocal of S/F) and h the tray spacing (in mm). u is a unit step function described as:

$$u = 0 \text{ for } (af) < 3.75 \text{ (mixer-settler regime)} \tag{5}$$
$$= 1 \text{ for } (af) \geq 3.75 \text{ (dispersion regime)}$$

The above correlation was tested for the other two flow ratios using a tray spacing of 100 mm and the results are shown in fig. 8 (unfortunately insufficient data was obtained to test the correlation for the 200 mm tray spacing).
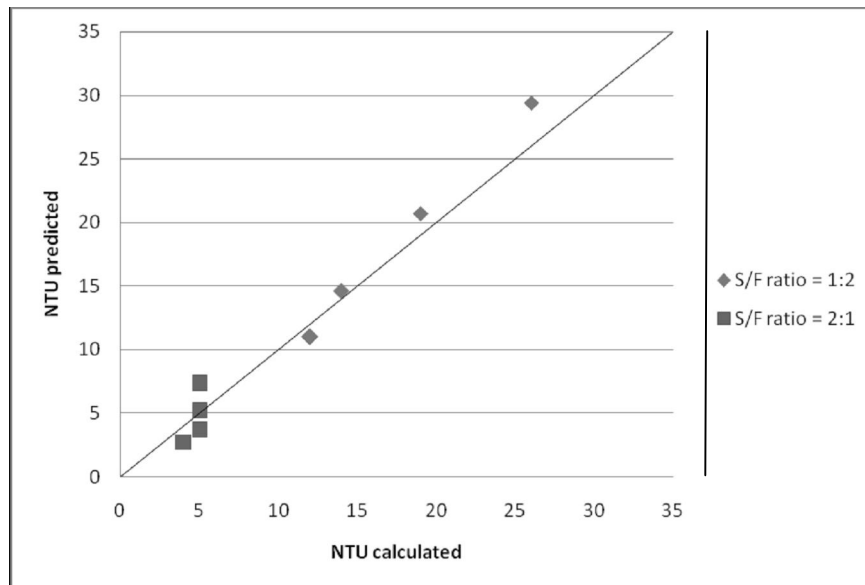
Fig. 8 – *NTU* model verification

The 45° line above shows a perfect relation between the model prediction and the actual values. As can be seen, the correlation offers a close approximation of the actual values with a maximum error of 6%.

Calculation of HTU

The height of a transfer unit was simply calculated from the following equation [13]:

$$HTU = {}^H\!/_{NTU}$$
$$(6)$$

with *H*, the height of the column being taken as 4.76 m. If we use the measured number of transfer units in the above equation, we will obtain the height equivalent to a theoretical stage (*HETS*) shown in fig. 9.
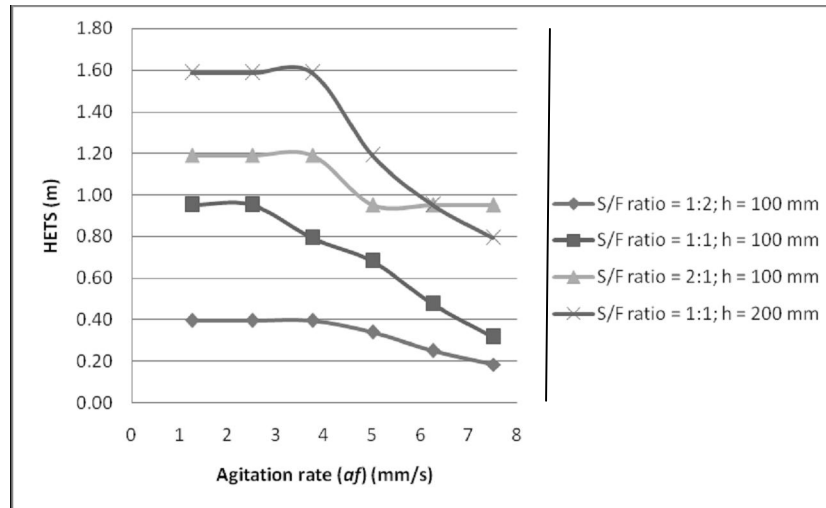
Fig. 9 – Effect of agitation level on *HETS*

The correlation for the prediction of *NTU* may be used for the prediction of *HETS* by replacing *NTU* by *H/HETS*.

$$\frac{H}{HETS} = \left[5.5 + \left(1.8e^{0.28(af)} - 5.5\right)u\right]L\left(\frac{100}{h}\right) \tag{7}$$

Mass Transfer Coefficient

The interfacial area of a drop distribution is related to holdup and d_{32} by [6]:

$$a = \frac{6\varnothing}{d_{32}} \tag{8}$$

and the true *NTU* is related to the interfacial area by [21]:

$$N_{ox} = \frac{k_{ox}aH}{U_d} \tag{9}$$

As a result the true *NTU* and mass transfer coefficient may be related to holdup and Sauter mean diameter by [18]:

$$k_{ox} = \frac{N_{ox} U_d}{aH} = \frac{N_{ox} U_d d_{32}}{6\emptyset H}$$

(10)

N_{ox} is fixed for a given tray spacing ($N_{ox} = 46$ for $h = 100$ mm; $N_{ox} = 23$ for $h = 200$ mm). U_d is also fixed for a given flow ratio and d_{32} and Ø were calculated above for different agitation rates. As a result it is possible to develop a correlation for the prediction of mass transfer coefficient for varying agitation rates and plate spacing. Fig. 10 show the results for the 2 tray spacings for the 1:1 S/F flow ratio.
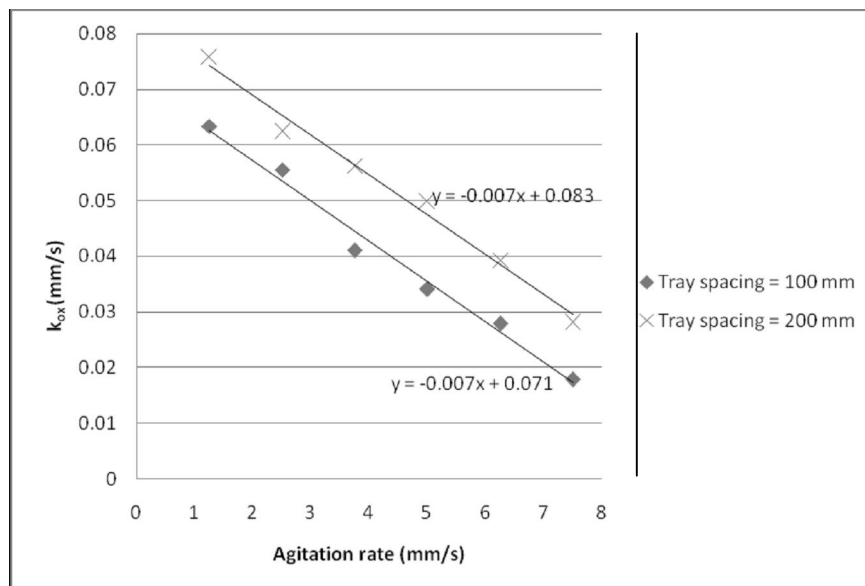

Fig. 10 – Mass transfer coefficient correlation

A general correlation for the evaluation of mass transfer coefficient was developed by combining the two equations above to give one correlation for the different tray spacings. This correlation is shown below where agitation rate and mass transfer coefficient are in mm/s and tray spacing is in mm:

$$k_{ox} = -0.007(af) + \left(0.06 + \frac{h}{10000}\right)$$

(11)

The correlation essentially only applies to the dispersion and emulsion regimes since the equation used to calculate the interface area involves the holdup of a drop dispersion. In the mixer-settler regime the holdup calculated includes the coalesced layer accumulated under the plates.

The above correlation was tested for the other two flow ratios using a tray spacing of 100 mm and the results are shown in fig. 11 (unfortunately insufficient data was obtained to test the correlation for the 200 mm tray spacing). Once again the 45° line shows a perfect relation between the model prediction and the actual values. As can be seen, the correlation offers a close approximation of the actual values with a maximum error of 13.4 %.
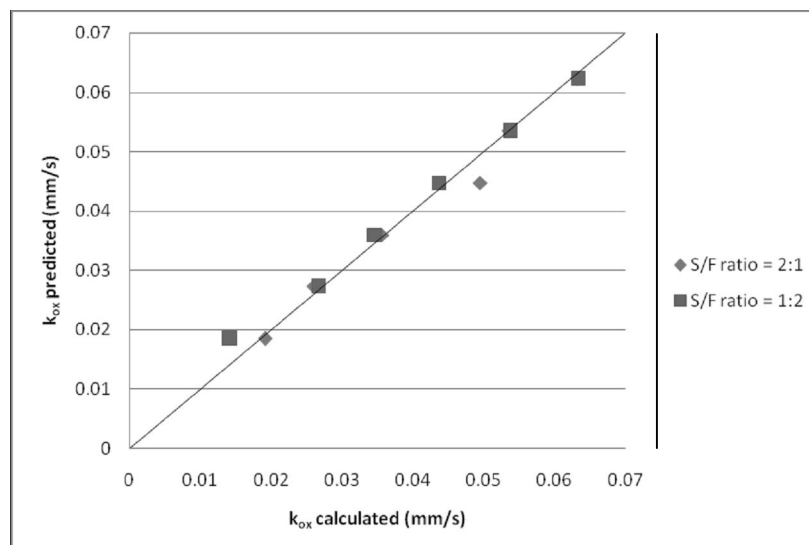

Fig. 11 – Mass transfer coefficient model verification

4. Conclusion

The ideal number of transfer units taking into account the backmixing of the phases was calculated graphically using the McCabe Thiele method. Using the information from *NTU* calculations an empirical correlation was developed for the prediction of the measured *NTU* taking into account axial dispersion (N_{oxm}). The data for the 1:1 flow ratio with both tray spacings was used to develop the correlation for the effects of agitation level, tray spacing and flow ratio. The correlation was tested with data for the other two flow ratios and showed reasonable accuracy with a maximum error of 6 %. The correlation was also adapted to calculate the *HETS*.

From the holdup and drop size data and the actual number of transfer units identified in the column, a correlation was developed for the prediction of mass transfer coefficient for the 1:1 flow ratio at both tray spacings as a function of agitation level and tray spacing. The correlation was tested for the other 2 flow ratios and showed reasonable accuracy with a maximum error of 13 %. The mass transfer coefficient was found to be independent of flow ratios.

Nomenclature

A	= amplitude (half-stroke), m
d_i	= dispersed phase drop diameter, m
d_{32}	= Sauter mean drop diameter, m
F	= frequency, Hz
H	= centre to centre plate spacing, mm
H	= height of the active part of the extractor, m
$HETS$	= height equivalent to a theoretical stage, m
HTU	= height of transfer unit, m
k_{ox}	= overall mass transfer coefficient, m/s
L	= ratio U_d/U_c
M	= slope of the equilibrium curve
n_i	= number of drops
N_{ox}	= 'true' overall number of transfer units based on the x phase
N_{oxm}	= measured overall number of transfer units based on the x phase
N_{oxp}	= apparent or piston number of transfer units assuming plug flow
NTU	= number of transfer units
S_i	= surface area of a drop, m^2
U_d	= superficial velocity of dispersed phase, m/s
X	= mol fraction in raffinate phase
Y	= mol fraction in extract phase
ϕ	= dispersed phase holdup

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