Pak. J. Sci. Ind. Res. 2006 **49**(1) 19-22

A Comparative Study of the Transient Response Characteristics of Laboratory-Scale Spray Columns and Packed Columns

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(received February 12, 2004; revised December 21, 2005; accepted January 3, 2006)

Abstract. Transient response characteristics of laboratory-scale spray columns and packed columns were studied by using impulse input tests. Using the results of impulse inputs, the dimensionless E-curves (normalized E-curves) were constructed and the variance (σ^2) of the exit age distribution function (E), and the dispersion number (D/uL), of the columns were computed using the dispersion model. This approach was useful for expressing the extent of mixing quantitatively, as well as evaluation of their comparative performance. It has been found that the dimensionless E-curves of the columns lie between plug flow and perfect mixing and the mixing pattern, whereas the extent of mixing in both spray and packed columns was found to be independent of the lengths of the columns. Nevertheless, the extent of mixing in the packed columns was much greater than in the spray columns.

Keywords: liquid-liquid extraction, spray columns, packed columns, transient response, exit age distribution, dispersion number, stimulus response, tracer input technique

Introduction

Liquid-liquid extractors can be theoretically designed for two ideal flow conditions, namely, (a) plug flow and (b) perfect mixing. Early designs of liquid-liquid extractors were based on the assumption of plug flow. In practical cases, however, the actual characteristics can show major departures from the ideal conditions. Thus, one may approach the problem of finding the equipment size for dispersed flow by calculating the one based on plug flow, and then making a correction for non-ideality, which occurs due to a number of factors. These factors may include molecular diffusion and eddy diffusion caused by agitation of pulsation and radial or Taylor diffusion resulting from non-uniform velocity, or velocity profile effects and dead zones. A detailed knowledge of the flow effects is, therefore, of great importance for the designing of liquid-liquid extractors. An approach of determining the non-ideality of flow could be to find out as to how long individual molecules stay in the columns (Danckwerts, 1953). This may be predicted by evaluating the exit age distribution function (E) by adopting an experimental technique known as the "stimulus response technique" (Levenspiel, 1958; 1962). In such experimentations a disturbance is given at the inlet of the system and then the corresponding response of the stimulus is recorded at the outlet. Out of the several experimental techniques known as stimulus response technique or tracer input technique, the impulse input technique is the simplest, which has been employed in the present investigation for the evaluation of performances of the spray and packed columns using water as the continuous phase and acetic acid as the tracer. The extent of non-ideality of flow has been accounted for as the dispersion number D/uL, the reciprocal of Peclet number, where D is the longitudinal or axial diffusion coefficient and u is the flow rate of the continuous phase and L is the length of column.

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Theoretical basis. From an equispaced time record of concentration of tracer at the outlet, the exit age distribution function E(t) is evaluated using the following relationship (Levenspiel, 1972).

$$E(t) = \frac{C}{\Sigma C \Delta t}$$

where:

C = the concentration of the tracer in the exit stream
t = is the sampling interval
The mean residence time($\bar{t}$) is evaluated as follows:

$$\bar{t} = \frac{\Sigma(tC)}{\Sigma C}$$

The exit age distribution function (E) in reduced time scale (θ) is given by:

$$E = \bar{t}\, E(t) = \frac{\bar{t}C}{\Sigma C \Delta t}$$

the mean age of the exit stream is:

$$\bar{\theta}_E = \bar{\theta}_C = 1 = \Sigma \theta_E \Delta\theta$$

and variance of the E distribution is:

$$\sigma^2 = \frac{\Sigma\theta^2 E}{\Sigma E} - 1$$

For a closed vessel, a finite vessel of length (L), the variance of E or C distribution is related to the vessel dispersion number as follows (Vogel, 1978):

$$\sigma^2 = 2\frac{D}{uL} - 2\left(\frac{D}{uL}\right)^2 (1 - e^{-uL/D})$$

As a first approximation, the second term on the right hand side was ignored to calculate the value of D/uL and a correction was then made by trial and error method.

The present work has been undertaken with the objective of comparing the transient response characteristics of laboratory-scale spray and packed columns so that the flow patterns and the extent of mixing in these columns can be determined.

Materials and Methods

Chemicals and materials used in the investigation. Acetic acid, 90% (Fluka Chemica, Japan); sodium hydroxide, 97% (Fluka Chemica, Japan); oxalic acid, 99.5% (E. Merck, India); phenolphthalein (E. Merck, India); Raschig rings (glass, dia 6 mm).

Determination of exit age distribution or residence time distribution in the extraction columns by the impulse input technique. The residence time distribution of materials in spray and packed columns of different lengths were determined by the impulse input technique (pulse test). For this purpose, distilled water was taken as the continuous phase by allowing it to flow at a constant rate through the inlet tip of the column. An aqueous solution of 1.049 g/ml of acetic acid was used as tracer. This solution (1 ml) was rapidly injected into the water stream at the inlet of the column. Samples were collected at the outlet of the column for a duration of 1 min each, at an interval of five min, and were analyzed for acetic acid by volumetric method with standard NaOH solution using phenolphthalein as indicator (Vogel, 1978).

Construction of spray and packed columns. Spray columns consisted of merely vertical shells with provisions for introducing and removing the liquids. Three spray columns (column I, II and III) were made from glass tubing of 2.54 cm internal dia having lengths of 20 cm, 40 cm and 60 cm, respectively. In order to obtain a continuous phase, the spray columns feed was allowed to be introduced from a 500 ml separating flask through a glass tip dipped in the fluid. The solvent was dispersed from another 500 ml separating flask through a stopcock fitted glass tip introduced at the bottom of the column. The use of different sizes of tips varied the flow rates of feed and solvent. The mouth of the separating flasks holding feed and solvents were fitted with dip tube ground joints so that constant rates of flow were maintained over a long period. This device was similar to the one used to control the flow-rate of solvent in flow microcalorimetry.

Results and Discussion

Responses to impulse inputs to laboratory-scale spray columns and packed columns of different lengths have been recorded using equispaced time (Table 1). The physical and statistical parameters of transient response characteristics of spray and packed columns using impulse inputs have been given in Table 2. From these results, concentration and time in reduced units were computed. Using these computed results dimensionless E curves have been plotted in Fig. 1 and Fig. 2, respectively, for the spray columns and packed columns.

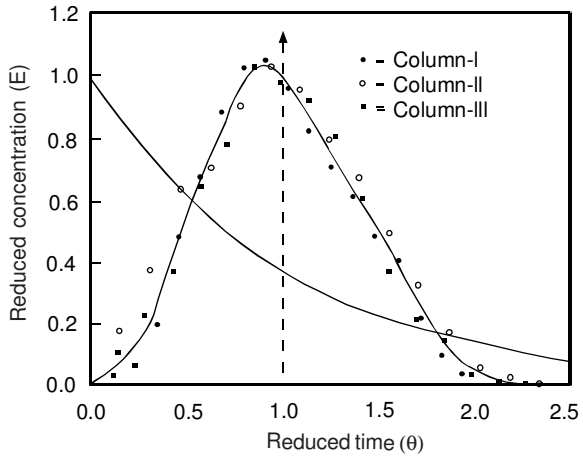

Fig. 1. Normalized exit age distribution function (E) curves for spray columns.

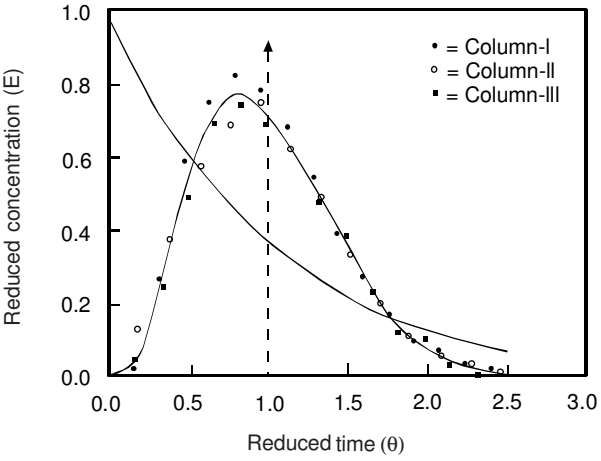

Fig. 2. Normalized exit age distribution function (E) curves for packed columns.

Theoretical curves for perfect mixing, the exponential curve, and plug flow have also been computed as dotted line, and have been incorporated in Fig. 1 and Fig. 2 for comparison. From Fig. 1 and Fig. 2 it may be observed that the experimental results lie between plug flow (no axial mixing) and perfect mixing. It may be further observed from Fig. 1 and Fig. 2 that there was a good deal of axial mixing or back mixing, both in the spray columns and packed columns. Following the dispersion model for closed vessels (Levensipel, 1962), the variances (σ^2) of the exit age distribution function (E) and the dispersion number (D/uL) have been computed.

It may also be noted from Fig. 1 that irrespective of the length of the spray columns, the E curves were almost super impossible giving variance of about 0.15, the corresponding dispersion number being 0.09. Similarly, it may be observed from Fig. 2 that the E curves for the packed columns were also superimposable giving variance of about 0.45 with a corresponding dispersion number of 0.25. A comparison shows

Table 1. Transient response characteristics of spray and packed columns using impulse inputs

Time (min)	Exit concentration of tracer, C (g/l)					
	Spray column			Packed column		
	column I	column II	column III	column I	column II	column III
5	0.12	0.90	1.10	0.13	1.20	0.77
10	0.22	1.96	2.50	1.43	3.41	4.18
15	0.68	3.36	4.20	3.25	5.17	8.69
20	1.70	3.78	7.60	4.16	6.27	12.10
25	2.40	4.76	9.00	4.55	6.83	13.11
30	3.10	5.44	11.80	4.29	5.62	12.21
35	3.60	5.04	11.20	3.77	4.41	10.24
40	3.70	4.20	10.60	2.99	3.00	8.27
45	3.40	3.60	9.20	2.14	1.88	6.74
50	2.90	2.60	7.00	1.52	0.98	3.99
55	2.50	1.68	4.20	0.96	0.56	2.12
60	2.20	0.84	2.40	0.59	0.30	1.70
65	1.70	0.28	1.60	0.42	0.09	0.56
70	1.40	0.11	0.40	0.20		
75	0.76	0	0.11	0.11		
80	0.32		0			
85	0.12					
90	0					

C = acetic acid used as tracer

Table 2. Physical and statistical parameters of transient response characteristics of spray and packed columns using impulse inputs

Physical and statistical parameters	Spray column			Packed column		
	column I	column II	column III	column I	column II	column III
Flow rate (ml/min)	6.8	6.2	2.6	5.66	4.38	1.66
Hold-up (ml)	296.5	198.4	91.6	176.5	115.3	59.6
Mean residence time (min)	43.6	32.0	35.2	31.81	26.31	30.25
Variance of residence time distribution (σ)	107.8	185.5	184.8	157.8	138.2	158.0
Initial tracer concentration (g/l, assuming ideal mixing)	3.54	5.29	11.45	5.51	9.09	17.60
Dispersion number (D/ul)	0.09	0.11	0.09	0.235	0.225	0.205

that the dispersion in the packed column was much greater than in the spray column.

It has been argued (Rahman *et al*., 1992) that for spray columns there are three distinct mechanisms of mass transfer, which are: (i) mass transfer during drop formation; (ii) drop movement and drop coalescence; and (iii) the mass transfer during drop movement in columns of short length, which is negligible. The present results appear to be independent of the length of the column showing that these were independent of the aforesaid mechanisms, probably because for the impulse input test a single continuous phase was present in the columns.

Conclusion

The observation that the exit age distribution (E-curves) both for spray and packed columns separately super impossible leads to the conclusion that the extent of mixing in spray and packed columns were independent of their lengths. However, comparatively higher values of dispersion numbers indicate that the extent of mixing in the packed columns was greater than that in the spray columns. It is further concluded that mixing in the spray and packed columns lies between plug flow and perfect mixing.

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