

## Tracer Density Effect on the Dynamic Liquid Hold-up in the Packed Distillation Column

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Tracer density effect on dynamic liquid hold-up in packed distillation column was investigated, with the view to explore the process hydrodynamics in design of reactive distillation in packed columns.

An increased dynamic liquid hold-up with tracer density rise was more pronounced at the lower segment of the packed section, notably for low percentage of aqueous methanol mixtures, to about 55 % increase for 0.1 mole fraction aqueous methanol mixture. The effect falls with increase in methanol concentration in the feed mixtures. The tracer concentration distribution across the packed section of the column supports backmixing of liquid phase in packed distillation operation, with dynamic liquid holdup concentrating at the lower part of the packed section.

Low pressure drop, associated with packed column, would be advantageous to reactive distillation operation in the column, especially for systems with high specific reaction rates.

*Key words:*

Tracer density, dynamic liquid hold-up, distillation

### Introduction

Although, the application of reactive distillation, which is an in-situ separation of process fluid, has increased rapidly in chemical and petroleum industries (Doherty and Maloney, 2001),<sup>1</sup> the design of columns for reactive distillation is yet to be fully explored. Kaymak and Luyben (2004)<sup>2</sup> itemized several limitations to effective application of reactive distillation. One of these limitations is the specific reaction rate of the system. To adequately cover this problem, consideration must be given to the residence time of species in the column, among other factors. This would require careful study of liquid hold-up in columns (especially packed columns) more so, that the residence time is an essential factor in chemical reaction – a criterion for reactive distillation. The hydrodynamics of this phenomenon requires understanding the liquid hold-up in beds and the residual liquid hold-up as a function of liquid flow and reactor geometry. Backmixing of phases, rather than plug flow model, had earlier been found adequate to describe concentration profile in packed column (Taiwo 1993,<sup>3</sup> Taiwo and Fasesan 2002<sup>4</sup>), but literature report on this phenomenon is sparse and hence the effect of dynamic liquid hold-up on its effectiveness and efficiency is not available. On this premise, this research work was designed to study the effect of the molecular species on the residence time in the reactor and hence the significance of the dynamic liquid hold-up operation.

### Experimental

The experimental rig (apparatus) (Figure 1) consists of a 0.1m internal diameter by 1.7 m long section of borosilicate pipe packed with ceramic

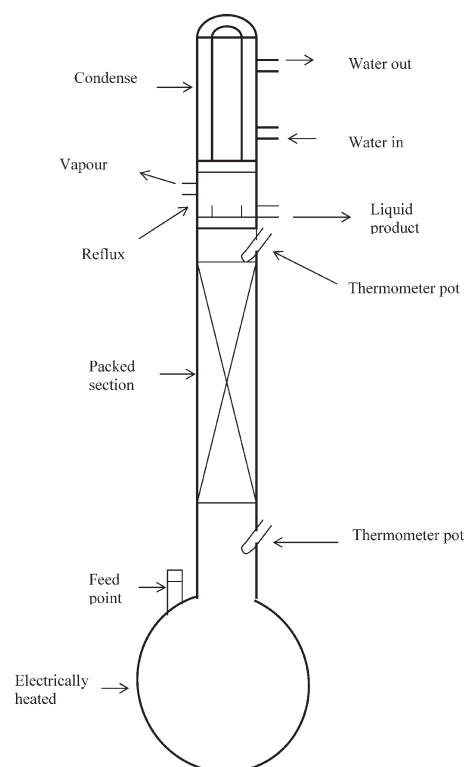


Fig. 1 – The Experimental Packed Distillation Column

Raschig rings (8 mm nominal diameter, and 12 mm length with  $492.05 \text{ m}^2 \text{ m}^{-3}$  volumetric area) randomly arranged in the column. Probes were introduced at three locations in the column to adequately measure the tracer distribution and the dynamic liquid hold-up.

The boiler pot was charged with aqueous methanol and the condenser unit was supplied with cooling water. The column was operated at atmospheric pressure and total reflux condition to attain steady state. On attainment of steady state, a situation indicated by constant column temperature across the column, the volume of liquid held up was determined from the height of the liquid held-up, read from the graduated scale fixed on the column at each of the designated segment of the packed column fixed with the probes. Thereafter, tracer sample which is 10 ml of  $10 \text{ g L}^{-1}$  KCl solution was injected into the column, through either the top or bottom port. The tracer distribution across the column was monitored by conductivity measurement. The probe and circuit for detection and transmission of conductivity measurement were designed as described by *Trasi and Khang* (1979).<sup>5</sup> A total of thirty-five experimental run were carried out and six of the experiments were repeated for consistency and reproducibility.

## Results and discussion

The tracer concentration profiles measured at the top and bottom-sampling points as a function of time are reported in figures 2 and 3. Figure 2 shows the distribution of tracer introduced into the column via the top port. A sharp rise in tracer concentration was observed at the top sampling point while a gradual rise was felt at the bottom sampling point. The concentration registered at the bottom point later rise above that of the top point. From figure 3, the changes in concentration of tracer across the column when injected at the bottom port was instantly felt at the bottom sampling point, and rose sharply with time, whereas at the top, the tracer was initially unnoticed until about 600 s. Even then, it was minimal (about  $35 \text{ mg L}^{-1}$ ).

The tracer concentration profiles in figures 2 and 3 reflect backmixing in the column. The trend was the same for all the experimental runs.

Table 1 shows that the dynamic liquid hold-up is resident mainly at the bottom section of the column. At the probe P1 (top section of the column) the measured hold-up for a 0.2 mole fraction aqueous methanol (run 8) was  $0.0243 \text{ m}^3 \text{ m}^{-3}$  as compared to the bottom probe P3 that had a dynamic liquid hold-up of  $0.0301 \text{ m}^3 \text{ m}^{-3}$ . This corroborates the earlier report of *Taiwo and Fasesan* (2004).<sup>6</sup>

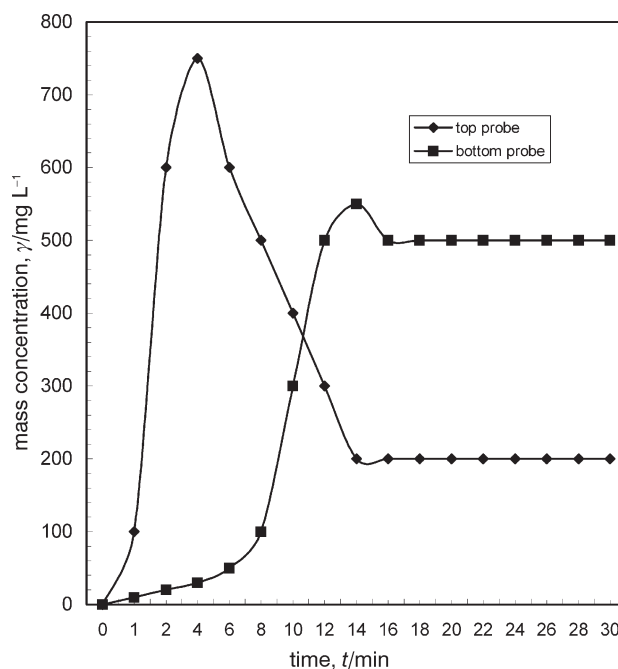


Fig. 2 – Tracer concentration distribution across the column packed section for tracer injected at the top port. (run 8)

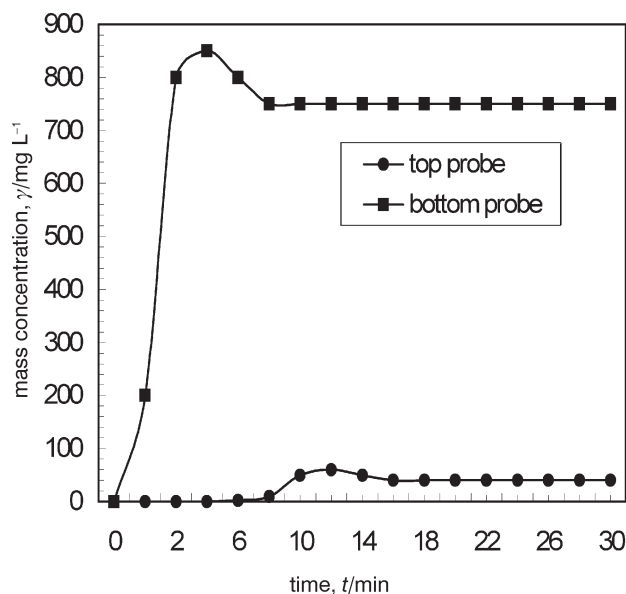


Fig. 3 – Tracer concentration distribution across the column packed section for tracer injected at the bottom port. (run 8)

Both table 1 and figures 2 and 3 reveal a continuous refreshing of the liquid held-up across the column as against the static holdup, hence the dynamic fraction of the liquid hold-up preponderates. This gives credence to backmixing in the packed distillation column operation. The dynamic nature of the liquid hold-up is responsible for the observed tracer concentration distribution. The highest tracer concentration was observed at the bottom sampling

Table 1 – Experimental dynamic liquid hold-up data

Run	Feed composition $x_{F,Me}$	Heat flow rate	Dynamic liquid hold-up, $H/m^3 m^{-3}$		
		$Q/kJ s^{-1}$	P1	P2	P3
1	0.1	0.195	0.0196	0.0228	0.0250
2	0.1	0.235	0.0204	0.0239	0.0269
3	0.1	0.260	0.0214	0.0245	0.0286
4	0.1	0.326	0.0229	0.0251	0.0294
5	0.2	0.195	0.0203	0.0232	0.0243
6	0.2	0.235	0.0210	0.0239	0.0272
7	0.2	0.260	0.0229	0.0244	0.0289
8	0.2	0.326	0.0243	0.0262	0.0301
9	0.4	0.195	0.0220	0.0248	0.0274
10	0.4	0.235	0.0245	0.0262	0.0290
11	0.4	0.260	0.0258	0.0289	0.0315
12	0.4	0.326	0.0273	0.0294	0.0322
13	0.6	0.195	0.0245	0.0260	0.0306
14	0.6	0.235	0.0252	0.0276	0.0314
15	0.6	0.260	0.0288	0.0304	0.0330
16	0.6	0.326	0.0305	0.0323	0.0351

point with highest dynamic liquid hold-up irrespective of the port of tracer injection. The tracer concentration, spread across packed section, was similar to the dynamic liquid hold-up distribution. This is an indication of favorable performance of packed column for reactive distillation, specifically, for systems with medium to high specific reaction rates: a condition requiring low pressure drop provided by packed column. Also, it suggests that, locating the reactants' introduction into the column at

a point a little above the reaction zone will probably give a more efficient design. This phenomenon will be explored in a future paper.

The influence of the tracer density variation was significant for the lower percentage aqueous systems. A 50 % increase in the tracer density resulted into increased dynamic liquid hold-up from 0.0243 to 0.0341  $m^3 m^{-3}$  at the top probe, and 0.0301 to 0.0466  $m^3 m^{-3}$  at the bottom probe for 0.2 mole fraction aqueous methanol (Table 2). This rise is

Table 2 – Tracer density influence of dynamic liquid hold-up

Run	Feed composition ( $x_{F,Me}$ )	Dynamic liquid hold-up $H/m^3 m^{-3}$					
		tracer density, $\rho = 10 g L^{-1}$			tracer density, $\rho = 15 g L^{-1}$		
		P1	P2	P3	P1	P2	P3
4	0.1	0.0229	0.0251	0.0294	0.0323	0.0374	0.0456
8	0.2	0.0243	0.0262	0.0301	0.0341	0.0387	0.0466
12	0.4	0.0273	0.0294	0.0322	0.0338	0.0371	0.0418
16	0.6	0.0305	0.0323	0.0351	0.0314	0.0332	0.0363

between 40 and 55 percent increase in the dynamic liquid hold-up. This could have resulted from either the cooling effect of tracer material resulting into ease of condensation of the vapor phase in a similar manner to cyclic cooling of vapor in distillation column presented by *Baron et. al.* (1980),<sup>7</sup> or increased molecular interaction of the molecular species resulting into drag action emanating from the coupling of molecules put forward by *Fasesan and Taiwo* (2001).<sup>8</sup> The increase was less than 5 % rise for the 0.6 mole fraction aqueous methanol studied. A dynamic hold-up of  $0.0314 \text{ m}^3 \text{ m}^{-3}$  at P1 and  $0.0363 \text{ m}^3 \text{ m}^{-3}$  at P3 was observed (run 16). This is approximately 3.0 and 3.5 percent rise, respectively. Therefore, the dynamic liquid hold-up of the separating mixtures having higher more volatile component (*mvc*) show less response to the tracer density increase. The tracer solution injected at the top is expected to cause more dispersion while that injected at the bottom would suppress dispersion, more so, that the tracer element are in upward and downward motion in the column, due to back mixing. Such effect would be felt at lower region than the upper region, since less dense tracer elements could be pushed upward more easily. Thus, exploring volatility for product separation in reactive distillation would be appropriate and efficient. The reactants species should be relatively heavier than the product to keep the reactant into the column. However, the distillate purity may depend on the conversion in the reactive zone. The experimental back-up of this will be explored in subsequent publication.

One significant observation is the fall in the dynamic liquid hold-up with increasing *mvc* of the separating mixtures fed into the column when highly dense tracer material was introduced. This is conspicuous in table 2. With the introduction of tracer solution with density  $15 \text{ g L}^{-1}$ , the dynamic liquid hold-up rose from  $0.0456 \text{ m}^3 \text{ m}^{-3}$  to  $0.0466 \text{ m}^3 \text{ m}^{-3}$ , and then fall to  $0.0418 \text{ m}^3 \text{ m}^{-3}$  and  $0.0363 \text{ m}^3 \text{ m}^{-3}$  for separating mixtures with 0.1, 0.2, 0.4 and 0.6 mole fractions aqueous methanol, respectively. This trend was consistent irrespective of the sampling location in the column and the boil-up rate.

## Conclusion

Dynamic liquid hold-up variation with tracer density in a packed distillation column has been evaluated. A significant change was found when the tracer density was increased by 50 percent for 0.2 mole fraction aqueous methanol mixture, and the effect dropped with increased *mvc* in the mixture.

The experimental data presented and analyzed, demonstrated that the hydrodynamic behavior of packed distillation column could effectively translate the reactive distillation process in packed column, which favors systems with high specific reaction rates.

## List of symbols

$a$	– volumetric area, $\text{m}^2 \text{ m}^{-3}$
$H$	– dynamic liquid hold-up, $\text{m}^3 \text{ m}^{-3}$
$a$	– heat flow rate, $\text{kJ s}^{-1}$
$t$	– time, min
$x$	– mole fraction
$\gamma$	– mass concentration, $\text{mg L}^{-1}$
$\rho$	– tracer density, $\text{g L}^{-1}$
$T$	– volume fraction, $\text{mL L}^{-1}$

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