# THE EFFECT OF MALDISTRIBUTION ON SEPARATION IN PACKED DISTILLATION COLUMNS

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#### ABSTRACT

For a packed bed in a distillation column,  $f_{max}$  is the maximum fractional liquid maldistribution that can be tolerated in a parallel column model whilst still being able to achieve the design separation. It was previously shown that  $f_{max}$  can be easily calculated from a conventional column simulation output and it is a measure of the sensitivity of a packed bed to maldistribution.

In this paper,  $f_{max}$  is applied to examples taken from air separation and ethylbenzenestyrene distillation. Using air separation plant data, it is shown that design separation shortfalls can be correlated against  $f_{max}$ . When  $f_{max} < 0.05$ , it is extremely difficult to achieve the design separation. A case study is given where  $f_{max}$  was increased from a very low value by splitting the bed, thereby achieving the design separation.

Application of  $f_{max}$  to ethylbenzene-styrene distillation leads to the conclusion that two and four packed beds should be used in the rectifying and stripping sections, respectively. By varying the number of stages in the beds in the stripping section to equalize the sensitivity to maldistribution, it is shown that it may be possible to use only three beds in future designs.

#### INTRODUCTION

Liquid or vapor maldistribution in packed distillation columns reduces the separation that is attained. Over the years, many workers have studied the problem in an attempt to predict the extent of maldistribution that occurs and to estimate its effect on separation. In a recent paper [1], the present authors introduced the concept of f<sub>max</sub> to characterize the sensitivity of a packed bed to maldistribution. To understand the significance of f<sub>max</sub>, consider the parallel column model shown in Figure 1 that represents liquid maldistribution in a packed bed. One side of the packed bed, represented by the left column in Figure 1, has a liquid flow rate of (1+f)L and the other (1-f)L. Hence, f is a measure of the fractional liquid maldistribution. Because of the different operating line slopes in the two columns, the overall separation obtained from the two column system is less than is obtained if the liquid is equally distributed. A typical calculated result is shown in Figure 2 where the effective number of stages from the combined two-column system decreases as the maldistribution fraction f increases. Also shown on Figure 2 is the limiting case labeled fmax. It represents the maximum maldistribution that could possibly be allowed while still being able to achieve the required separation. For a fixed arbitrary value of the maldistribution fraction f (say 0.04), whether or not a given bed is sensitive to maldistribution depends only on the value of  $f_{max}$  for the bed. Thus, in Figure 2, a bed having 10 theoretical stages in each parallel column corresponds to the line that cuts the ordinate at N=10 when f=0. For this bed,  $f_{max} > 0.1$  from Figure 2 and if f=0.04, the combined bed actually delivers about 9.5 effective theoretical stages, so that the effect of maldistribution is negligible. On the other hand, a bed having 40 theoretical stages in each parallel column (for which f<sub>max</sub>=0.038) provides only 24 effective stages if f=0.04. Thus, the designer need only determine the value of f<sub>max</sub> for the desired separation that is required to characterize the sensitivity of the overall bed to maldistribution.



Figure 1. Parallel column model



Figure 2. Results from the parallel column model  $\alpha$ =1.538, y<sub>o</sub>=0.5, y<sub>N</sub>=0.95. x<sub>N+1</sub>=0.95

It was shown in reference [1] how  $f_{max}$  varies with the number of stages, relative volatility, terminal concentrations and molar liquid to vapor ratio. The following equation was derived for a binary system from which  $f_{max}$  can easily be calculated.

$$f_{max} = \frac{y_{N+1} - y_N}{y_N - y_o} + \frac{x_1 - x_o}{x_{N+1} - x_o} - \left(\frac{y_{N+1} - y_N}{y_N - y_o}\right) \left(\frac{x_1 - x_o}{x_{N+1} - x_o}\right)$$
(1)

The concentrations involved are shown on Figure 1, where in addition,

$$y_{N+1} = \frac{\alpha x_{N+1}}{1 + x_{N+1}(\alpha - 1)}$$
(2)

and

$$x_{o} = \frac{y_{o}}{y_{o} + \alpha(1 - y_{o})}$$
(3)

Note that it is not necessary to construct a parallel column model to determine  $f_{max}$  from equation (1) and the concentrations required are usually readily available from the output of a conventional column simulation program.

In the present paper, we apply the concept of  $f_{max}$  to two important industrial separations that are carried out by distillation using packed columns namely, air separation and ethylbenzene-styrene separation.

#### **AIR SEPARATION**

A typical three-column system used in air separation is shown in Figure 3. Saturated air is fed to the bottom of the high pressure lower column 1. Condensation of nitrogen at the top of the lower column is used in a reboiler-condenser to generate oxygen vapor for the low pressure upper column 2. Nitrogen and oxygen products are taken from the top and bottom of the upper column, respectively. A side stream is taken from part way up the upper column that contains 5-15% molar argon. This stream is fed to the argon column 3 from which an argon stream is removed as an overhead product. Condensation at the top of the argon column is provided by boiling a liquid stream taken from the bottom of the high pressure lower column 1.



Figure 3. Three column system for air separation

The internals of the three distillation columns involved can consist of trays, packing (typically structured) or a combination of both. It is evident from Figure 3 that a number of distinct column sections are involved, particularly in the upper column, because of the existence of multiple feeds and draws. In addition, when packing is the internal of choice, a given column section is often subdivided into two or more packed beds using liquid collectors and redistributors between the beds. In the argon column, when very pure argon is produced by distillation directly (oxygen <5 ppm),

about 180 theoretical stages are needed. It is then usual to have upwards of 10 beds of packing in the argon column stacked one above the other.

Structured packing has been used in Praxair's air separation columns for more than 15 years and there are many hundreds of packed beds in operation. The great majority of these beds operate at or better than design. However, there have been a comparatively small number of beds which, on start-up, gave a separation that was initially less than design. In most cases the discrepancy was minor and no action needed to be taken, but in a few others remedial action was necessary.

A survey was undertaken of those beds for which the separation performance was initially less than design. At the same time, the value of  $f_{max}$  was calculated for those beds using equation (1) in conjunction with an appropriate column simulation. The results are shown in Figure 4 as a plot of the measured theoretical stages as a percent of design versus  $f_{max}$ .



Figure 4. Air separation plant data for packed beds where a separation shortfall was observed

The data shown on Figure 4 can be arbitrarily divided into four bands. When  $f_{max} > 0.20$ , the beds are insensitive to maldistribution. The small number of apparent theoretical stage shortfalls can be readily explained by composition sampling uncertainties between beds and by rounding the number of theoretical stages in a bed to an integer.

When 0.10 <f<sub>max</sub> <0.20, the beds are a little more sensitive to maldistribution but theoretical stage shortfalls from maldistribution in this range of f<sub>max</sub> only occur when the maldistribution is very severe. As an example, in one case significant maldistribution was traced to a leaking liquid collector, which then caused a minor separation shortfall.

When  $0.05 < f_{max} < 0.10$ , packed beds are quite sensitive to maldistribution and although the design separation can usually be obtained, the potential exists for a

significant shortfall in the theoretical stages obtained unless the quality of distribution is very good.

When  $f_{max}$  <0.05, the theoretical stage shortfall can be very substantial and it can be difficult to achieve the design separation even with very uniform distribution.

The following describes a case history of a plant that, on start-up, did not perform satisfactorily because of poor performance of the packed bed labeled A in Figure 3. The concentration of nitrogen in the liquid entering this bed was 44% molar. The mixture was assumed to be a pseudo-binary, where oxygen and argon were lumped together as one component. The design concentration for nitrogen in the liquid leaving the bed was 17 ppm molar. The latter concentration is important because all the nitrogen that enters the argon column 3 in the feed from the upper column 2 ends up in the argon product, in the absence of a pasteurization zone at the top of the argon column feed, bed A was designed to deliver 15 theoretical stages. The design value of  $f_{max}$  for the bed was 0.008. However, on initial start-up, only 11 theoretical stages were achieved with the result that the nitrogen concentration in the argon product was unacceptably high.

The diagnosis was that the shortfall was caused by the combination of the inevitable small amount of liquid maldistribution that existed in bed A together with the very low value of  $f_{max}$ . The remedy was to reduce  $f_{max}$  by shortening the packed beds.

The plant was shut down and bed A was replaced by two beds designed to deliver 7 and 8 theoretical stages, respectively. A new collector-redistributor was installed between the beds to mix and redistribute the liquid. On restart, the combination of the two beds achieved the design number of theoretical stages and the nitrogen content of the argon product was within specification. The calculated values of  $f_{max}$  were 0.11 for the upper bed and 0.51 for the lower bed, which were therefore significantly increased over the original value.

The importance of using two packed beds of about equal height in this section of the upper column has been noted previously [2].

### ETHYLBENZENE-STYRENE DISTILLATION

Separation of ethylbenzene from styrene is an important vacuum distillation operation that is widely practiced. Table 1 summarizes the key parameters used in the present study. The column was simulated with the commercial process simulator Hysys, using the Wilson property package. The concentration profiles that resulted from the simulation involved the four components indicated in Table 1. They were converted to a pseudo-binary involving only ethylbenzene and styrene by dividing by the sum of the ethylbenzene and styrene mole fractions. The pseudo-binary concentration profiles were then used to calculate  $f_{max}$  from equation (1).

Two cases were considered for the rectifying section that contains 26 stages: a single bed and two beds of 13 stages each. Four cases were considered for the stripping

section that contains 69 stages: a single bed, two beds, three beds and finally four beds. As far as possible, the theoretical stages were allocated equally between beds.

Component	Feed wt %	Overhead wt %	Bottoms wt %
Lights (toluene)	0.5		
Ethylbenzene	35		75x10⁻⁴
Styrene	62.5	2.0	
Heavies (diethylbenzene)	2.0		
Feed rate	10 <sup>5</sup> kg/h		
Feed temperature	99°C		
Reflux ratio	8.0		
Top pressure	120 mbar		
Bottom pressure	213 mbar		
Theoretical Stages	26 rectifying section 69 stripping section		

Table 1. Ethylbenzene-styrene distillation

The results are shown in Figure 5 for the rectifying section. It shows that  $f_{max}$ =0.066 if one single bed of packing is utilized in this section. However, if the section is split into two beds, the value of  $f_{max}$  in each bed jumps to greater than 0.14. A rule of thumb, based on air separation experience (see also Reference 1), is that it is very difficult to achieve the design separation if  $f_{max}$  <0.05. However, if 0.05 <  $f_{max}$  < 0.10 the separation is possible but will be sensitive to maldistribution. For  $f_{max}$  >0.10 the bed will not be particularly sensitive to maldistribution. Based on the above it is evident that the prudent design is to use two beds in the rectifying section. Two beds are in fact used in practice.



Figure 5. Ethylbenzene-styrene rectifying section

The results for the rectifying section are shown in Figure 6. In Figures 5, 6, and 7 where more than one bed is used, the beds are shown in order with the left hand bar representing the top bed and the right hand bar representing the bottom bed. Figure

6 shows that not until four beds are used does the value of  $f_{max}$  for the bottom bed increase above the critical value of 0.05. This is in agreement with industrial practice where four beds are indeed usually used in this section. It is evident from Figure 6 that the use of an equal number of stages in each bed is not optimum. There seems to be an opportunity to use fewer stages in the bottom bed and rather more in the middle bed(s) of the section with the intention of achieving the same value of  $f_{max}$  in each bed.



Figure 6. Ethylbenzene-styrene stripping section Equal division of stages between beds

This is explored further in Figure 7 where three beds are used in the stripping section. As the number of stages in the bottom bed is progressively decreased from 23 to 14, the value of  $f_{max}$  for that bed increases to an acceptable value of 0.12. Similarly, it also seems preferable to reduce the number of stages in the top bed from 23 to 18. The final design has 18, 37 and 14 stages in each bed rather than the originally assumed equal split of 23, 23 and 23.



Figure 7. Ethylbenzene-styrene three bed stripping section Unequal division of stages between beds

The explanation for this finding can be found in the McCabe-Thiele diagram for the separation. The top and bottom of the stripping section are both relatively pinched, whereas the middle section is unpinched at both ends. Thus, the middle section is less sensitive to maldistribution and more theoretical stages can be used in that section.

By optimizing the beds in this way, it should be possible to achieve a column design that uses only three packed beds rather than the four beds currently used in practice, with consequent savings in the cost of column internals and column height.

The discussion above resulted in a design with nearly equal values of  $f_{max}$  in each of the three beds that make up the stripping section. Thus, each bed has approximately the same sensitivity to maldistribution. In recommending an optimized theoretical stage allocation between beds based on this, it is implicitly assumed that liquid maldistribution in a bed reaches an equilibrium value and does not progressively continue to increase as it flows down through the bed. Thus it is assumed that the sensitivity of a bed to maldistribution depends only on  $f_{max}$  and does not increase with bed length because of increasing maldistribution. The existence of a natural or equilibrium liquid maldistribution profile has been deduced by Albright [3], by Zuiderweg [4] and by Sun et al. [5], so there is ample justification for this assumption.

## CONCLUSIONS

It has been demonstrated that for any given packed bed having a design separation, it is possible to calculate the corresponding value of  $f_{max}$ . Using plant data from air separation, it has been shown that the shortfall in separation for beds that failed to meet design could be correlated with  $f_{max}$ . Beds having  $f_{max} < 0.05$  are particularly at risk of failing to achieve the design separation. It was shown that calculation of  $f_{max}$  for the beds in an ethylbenzene-styrene splitter indicated that two and four beds should be used in the rectifying and stripping sections, respectively. The use of  $f_{max}$  to optimize the theoretical stage split suggests that it may in fact be possible to use only three beds in the stripping section for future designs.

## NOMENCLATURE

- f liquid maldistribution fraction
- f<sub>max</sub> maximum value of f above which the separation cannot be achieved
- L liquid flow-rate, kg-mol s<sup>-1</sup>
- N effective number of theoretical stages
- N<sub>A</sub> actual number of theoretical stages
- V vapor flow-rate, kg-mol s<sup>-1</sup>
- x mole fraction of more volatile component in liquid
- y mole fraction of more volatile component in vapor
- $\alpha$  relative volatility

### Subscript

n leaving theoretical stage n

#### **Superscripts**

- ' for column 1
- " for column 2

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