

THREE-PHASE DISTILLATION IN PACKED COLUMNS: GUIDELINES FOR DEVELOPMENT, DESIGN AND SCALE-UP

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INTRODUCTION

Multi stage distillation processes are normally operated in columns, equipped with trays or packing to obtain counter current flow of vapour and liquid. In tray columns the liquid is moving from the inlet over the active area to the exit downcomer. The necessary mass transfer area between the phases is achieved by intense mixing of vapour and liquid on the bubbling area.

In packed columns the flow of both phases is directed by the packing itself. Liquid is wetting the packing surface and flowing downwards counter currently to the ascending vapour. The necessary mass transfer area is provided by high packing surface area.

In either case the substantial hydraulic characteristic of the different constructions is that the counter current flow of vapour and liquid is directed through the column internals. Internals such as dual flow commonly show instabilities in operation due to insufficient direction of the phases, especially when they are operated beyond the designed load point.

CHARACTERISTICS OF THREE-PHASE DISTILLATION

While the flow characteristics in tray columns remain the same with the existence of a second liquid phase, those of packed columns result in different flow patterns for the two liquid phases, due to their different wetting capabilities. Additionally in tray columns mass transfer between the two liquid phases is ensured by an intense mixing of the two liquid phases. Therefore, in industry, tray columns are widely used for the distillations of mixtures with miscibility gaps.

Typical pressure drop in tray columns is from 3 to 6 mbar per theoretical stage. Accordingly, separations which require a high number of stages and a small pressure drop cannot be accomplished in a tray column. For example, a separation that requires 50 or more theoretical separation stages and which, due to a thermally unstable product, cannot have a sump pressure higher than 150 mbar, cannot be performed in just one tray column.

The pressure drop problem can be solved with a packed column, in which a high number of stages with moderate pressure drop can be accomplished. However, the problems originated by the introduction of two liquid phases must be considered in development, design and scale-up.

DESIGN OF THREE-PHASE DISTILLATION IN PACKED TOWERS

In industrial practice the design of distillation processes is typically based on the equilibrium stage concept. This is only limited capable for systems with two liquid phases, since the different flow patterns result in different residence times of the individual phases.

Mass transfer models are basically capable of describing the relation of packed columns with two liquid phases. However in addition to the thermodynamic mixture properties, the mass transport phenomena must be investigated with a variety of parameters, which are generally difficult to generate [1].

A crucial part of sound distillation processes design, especially for new processes, are continual laboratory experiments in columns with a diameter smaller than 100 mm. Supported by model calculations the height of the packing and the necessary reflux ratio can be determined through the experiment for conventional as well as for three-phase packed column.

In order to maintain the same flow characteristics in the laboratory and on a technical scale, a similar type of packing should be used in the experiment and the technical implementations.

In a three-phase case heuristic knowledge, conventional model calculations and special constrains must be considered for the scale-up to technical dimensions.

SCALE-UP: DESIGN OF LIQUID DISTRIBUTORS FOR WITH TWO LIQUID PHASE APPLICATIONS

PROBLEMS OF CONVENTIONAL LIQUID DISTRIBUTORS

Essential for the performance of packed columns is a sufficient distribution of the liquid phase. In order to keep maldistribution in the packing at a minimum, the areas with large packing heights are divided into smaller segments. Between the segments the liquid is collected, mixed and distributed again onto the packing section below.

Trough distributors are often used as liquid distribution systems. The liquid flows directly from the feed pipe into a parting box where it is separated into the troughs and from there it is evenly distributed across the packed bed through small orifices in the bottom or slots on the sides of the trough. An even distribution of the liquid is ensured by a minimum liquid head above the orifices.

With the presence of a second liquid phase three different zones in the distributor can be observed. Above the clear heavy phase, which settles into the lower part of the distributor, there is a zone in which both phases form a cloudy layer. Above the cloudy layer is normally a zone where the lighter phase has settled. The height of the individual zones is dependent upon the separation characteristics of the phases and significantly influences the distributor performance.

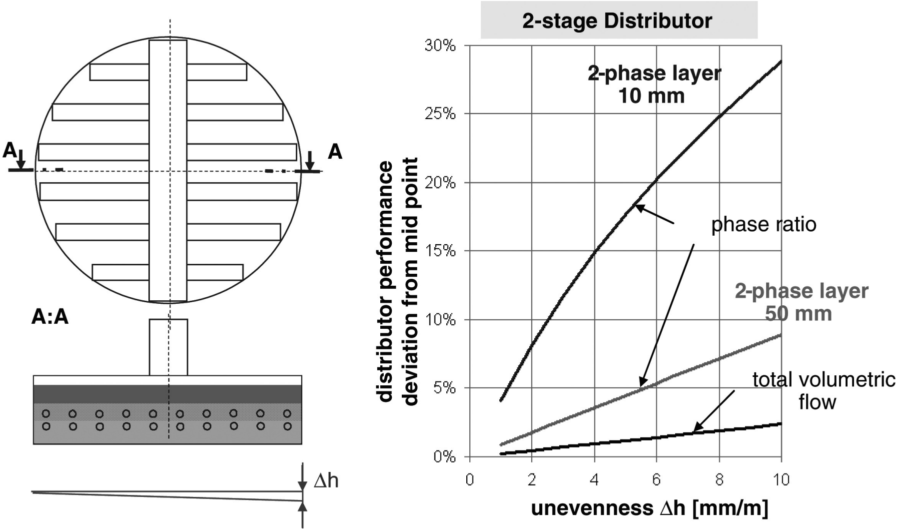

Figure 1. Distribution quality of a 2-stage distributor

TWO STAGE LIQUID DISTRIBUTOR

If for example a common trough distributor with two stages is considered as technical setup, the performance is strongly dependent on an even assembly. Figure 1 shows model calculations of the quality of the distribution as dependent upon the height of an unevenness Δh. Although the distribution quality of the total volumetric flow through the appointed troughs is minimally affected by the height of the unevenness, a resulting drastic dip in the distribution quality of the phase ratios can be observed. Besides the unevenness the distributor performance is strongly dependent on the height of the cloudy two phase layer and the performance is lower as the height of the cloudy two phase layer decreases.

THREE STAGE LIQUID DISTRIBUTOR

Improved quality of distribution for the phase ratio can be achieved by the use of a distributor with three vertical outlets.

The distributor has to be designed in a way that the cloudy two phase layer is located between the lower and the upper orifice. The top orifice is exclusively for the specific light phase discharge and the specific heavy phase flows through the bottom orifice. Since the top and bottom orifices are each designed for a homogenous liquid layer, moderate distributor unevenness has no influence on the phase fraction, which flow through the top and bottom orifice.

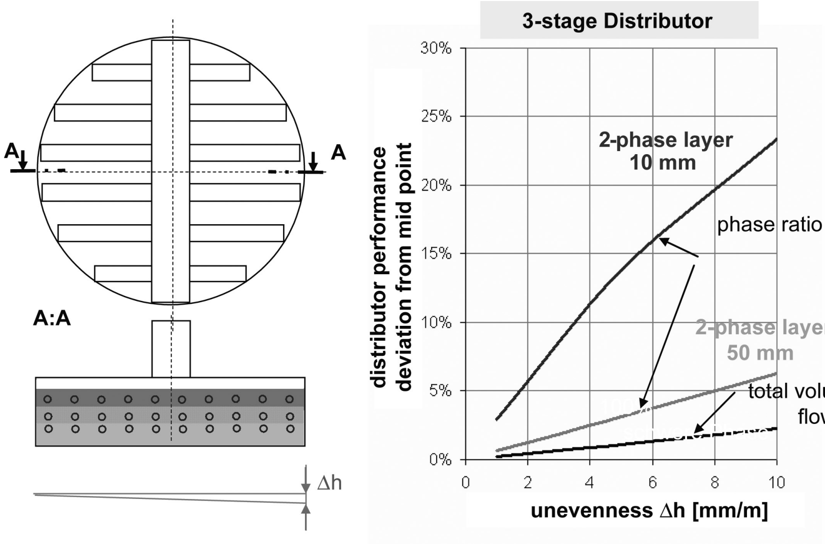

Figure 2. Three stage liquid distributor in two liquid phase applications

The middle orifice is arranged in the cloudy two phase layer, where phase ratios depend on the height coordinate. Therefore an uneven distributor will lead to different phase ratios at the individual orifices. To achieve good net distribution qualities (sum of all orifice flows) a minimized flow through the middle orifice is favourable. Unfortunately a distributor designed in this way is only feasible for high liquid loads.

CAPACITY OF PACKED COLUMNS WITH TWO LIQUID PHASES

Examinations on packed columns with two liquid phases have shown, that the presence of a second liquid phase lead to an increase in the pressure drop and therefore to a reduced operation window [2]. Higher pressure drop is caused by an increase in liquid hold-up. For mesh packing operated under two liquid phase conditions a good agreement between measured and calculated pressure drop can be achieved, when a hold-up is considered, which consists of the addition of the hold-ups of both individual liquid streams [2].

Figure 3 comprises model calculations for pressure drop in a packed column, operated with two liquid phases. Beneath the stagnation point the liquid hold-up is solely dependent upon the liquid load; the differences between two and three phase operations are negligible. Bigger differences between two and three phase operating methods are first noticeable at higher vapour rates and increase with higher liquid loads.

Column operation under vacuum usually leads to low liquid loads and therefore only to small pressure drop differences between two and three phase operation. The

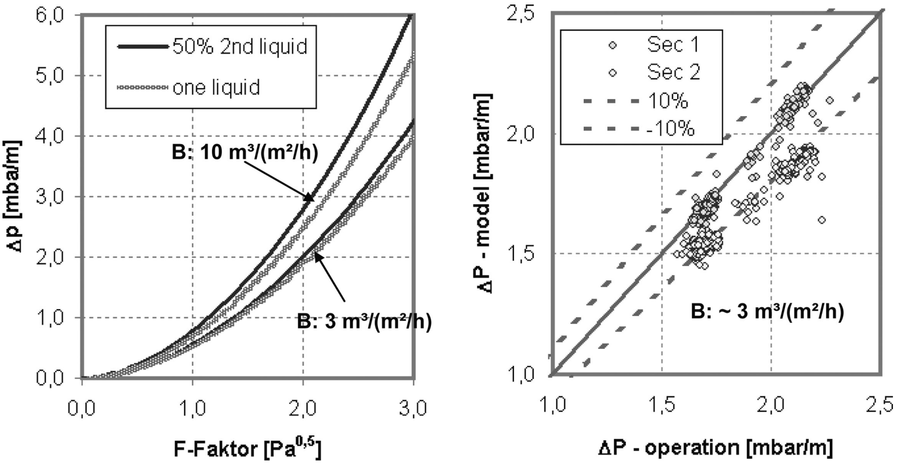

Figure 3. Pressure drop of three-phase distillation columns – model calculations for different liquid loads and a comparison to data points from a column in technical scale

design at low liquid loads can be based on conventional pressure drop calculation for two phase systems. The part to the right in Figure 3 compares model calculations to measured pressure drop in an industrial 3-phase column. The measured values are reproduced very accurately through the model.

CONCLUSION

The design of three phase packed columns requires the consideration of additional variables compared to conventional two phase applications. Equilibrium stage calculations, which are commonly used in industry for the thermodynamic design of distillation processes, are roughly applicable for the description of the behaviour with the introduction of two liquid phases.

An accurate design can result from continuous laboratory experiments, from which the height of the internal construction and the resulting reflux ratio can be determined. In order to maintain same flow characteristics in the laboratory and on a technical scale, the similar type of packing should be used in the experiment and the technical implementations.

A special liquid distribution system must be considered for the scale-up to the industrial diameter, in order to realize a sufficient distribution quality for the phase ratios.

The determination of the column diameter for vacuum columns, which are commonly used with low liquid load, can result from common pressure drop correlations.

LITERATURE

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