

Experimental Investigation of Foam in Packed Distillation Columns

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Abstract. Foam formation in distillation columns lead to various negative effects such as reduced distillation throughput and reduced separation performance. In order to determine the influence of foam on distillation throughput and separation performance, measurements with chlorobenzene/ethylbenzene mixtures in a DN50 laboratory distillation setup were carried out. This contribution specifically addresses the investigation of foam in structured packings in comparison to randomized packings with different materials. It is shown that structured packings were more susceptible to foam and showed greater losses in separation performance under influence of foam than randomized packings. However, in absolute terms, foam-loaded structured packings showed the highest separation efficiencies compared to the randomized packings.

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Introduction

Foam in distillation processes is one of the main disturbances in the operation of distillation columns in the chemical and food process industry [1,2]. Foaming issues are known from gas treatment, distillation [3] and extraction processes. Especially columns in CO₂ absorption with amine scrubbing are affected [4,5].

Commonly observed unwanted impacts corresponding to foaming are process impairments, high pressure drops, reduced throughput, lowered heat transfer and reduced separation performance. This leads to a reduction in process efficiency and increased specific production costs which can even result in production downtime [2].

Foaming can affect the capacity of fractionation units by hindering mass and phase separation and limiting the capacity of downcomers. Unwanted foams cause flooding of columns to be reached earlier and reduce separation capacities [6]. Especially in packed columns, foam can reduce mass transfer efficiency by about 20 % and packing capacity by about 30 % [2,7]. So far, there are only few systematic studies on the effects of foaming on fractionation equipment. Most studies on corresponding foams [7–9] have dealt with the effect of foaming on apparatus capacity. As yet, no models are published that can predict separation performance in foaming systems [7]. Therefore, so-called "de-rating factors" are used in the design of processes, susceptible to foaming. This is based on empirical values, e.g. on the performance of column internals. However, de-rating factors only apply to certain types of internals. For example, the empirical "system factor" was developed for tray columns and is not sufficient to describe the foam influence in packed columns [10]. Within the reboiler of distillation columns, evaporation-induced foam can occur and rise into the column and its separation internals. Herein foam often causes reduced fractionation efficiency and premature flooding [6].

In general, foam can result from the presence of surface-active components that accumulate at the interface between the liquid and vapor phases [11], or can be caused by miscibility gaps (Ross-type foaming) [12]. Foam-stabilizing surface-active components can often represent impurities such as decomposition compounds or particles in industrial processes [11,13–15]. In this paper, evaporation-induced foam formation based on the presence of impurities like particles is investigated.

Experimental setup and operation

A laboratory packed distillation column (Fig. 1) was set up to investigate the distillation throughput and the separation performance under foam conditions. The column with an inner diameter of 50 mm was packed with three different packing materials and operated in total reflux mode [3].

An electrically heated (700 W; PILZ[®] Classic-Line G2, Winkler AG, Heidelberg/Germany) 2000 mL glass flask is assembled with a double-walled glass cylinder with the dimensions d x H = 50 x 800 mm (QVF Engineering GmbH, Mainz/Germany). To support constant and defined boiling, 2 g boiling chips (B2, Pöllath Labortechnologie,

Bamberg/Germany) were added to the flask. The double-walled column was heated with oil. The thermal oil "Renolin Therm 320 Labothermol® O" (neoLab Migge GmbH, Heidelberg/Germany) was pumped through the "heating jacket" by a thermostat (HUB_16005, Peter Huber Kältemaschinenbau AG, Offenburg/Germany). The oil temperature in the heating jacket was set to the boiling temperature of the light boiling component chlorobenzene (CB) ($T_{oil} = 132\text{ °C}$). The column was operated at ambient pressure (1 bar abs), as a constant foaming capacity was achieved at this pressure. With this setup an adiabatic foam generation and growth could be approximated which reflects typical conditions in thermal separation plants. The evaporation-induced foaming ability in a technically relevant operational mode of distillation columns with infinite reflux and a correlation between evaporation-induced foam and separation performance as well as distillation throughput may be realized.

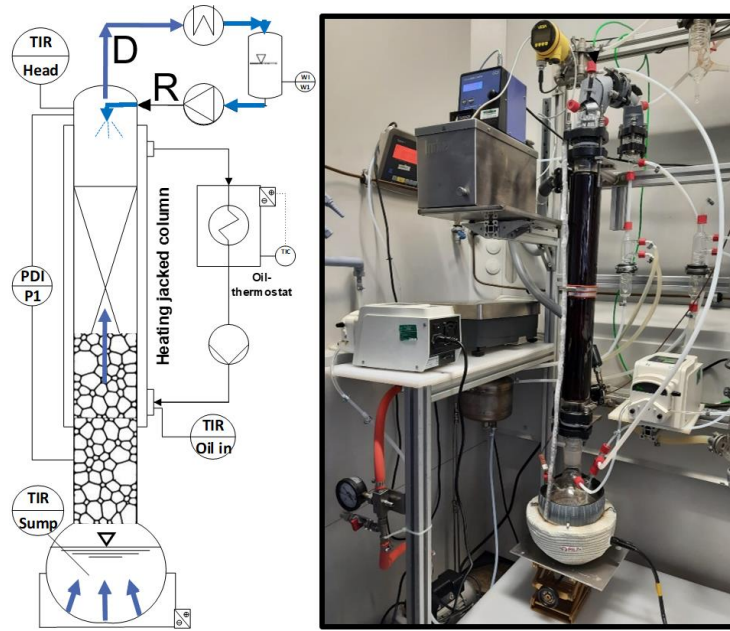


Fig. 1. Schematic diagram (left) and photo (right) of distillation setup with the sumps heating mantle and heated DN50 column.

The foam produced by evaporation in the sump rises through the gas flow up into the oil-heated column. The foam formation depends on the gas load factor f_G , which is calculated according to Eq. 1.

$$f_G = \overline{w_G} \cdot \sqrt{\rho_G} = \frac{(w_{G, \text{sump}} + w_{G, \text{head}})}{2} \cdot \sqrt{\rho_G} = \frac{(\dot{V}_{G, \text{sump}} + \dot{V}_{G, \text{head}})}{2 \cdot A_{cs, col}} \cdot \sqrt{\rho_G} \quad (1)$$

$$= \left(\frac{\dot{Q}_{\text{prod}}}{h_v \cdot \rho_{G, \text{sump}}} + \frac{\dot{m}_{G, \text{head}}}{\rho_{G, \text{head}}} \right) \frac{\sqrt{\rho_G}}{2 \cdot A_{cs, col}}$$

The gas load factor f_G was calculated based on the arithmetic mean of the gas velocities w_G in the head and sump of the column. The gas velocities relate to the gas flow through the cross-sectional area of the column $A_{cs, col}$. The gas volume flow below the packings $\dot{V}_{G, \text{sump}}$ was determined using the heat flow transferred to the product $\dot{Q}_{\text{prod}}$ in the evaporator. The gas volume flow above the packings $\dot{V}_{G, \text{head}}$ was determined using the gravimetrically measured condensate mass flow rate. The vapor density ρ_G of the processed mixture was approximated using the ideal gas law and Amagat's law [16].

The distillate vapor flow is condensed in a glass heat exchanger. The condensate is collected in a non-heated container and returned as reflux stream at the top of the column. When a steady state is reached, separation performance measurements were conducted. The steady state operational mode is necessary for investigations of separation efficiencies [7] and was ensured in this setup via constant mass flows.

In the total reflux mode, the distillate flow $\dot{V}_D$ corresponds to the reflux flow $\dot{V}_R$ at the head of the column. Therefore, the liquid load w_L of the reflux stream according to Eq. 2 is used to describe the distillation throughput.

$$w_L = \frac{\dot{V}_D}{A_{cs, col}} = \frac{\dot{V}_R}{A_{cs, col}} \quad (2)$$

The foam volume V_{foam} in the distillation section, consisting of the column, piston and an intermediate part, is represented by the foam capacity c_F . The foam capacity c_F quantifies the capacity of a system against foam and corresponds to the free volume V_{free} , which is not occupied by foam, see Eq. 3.

$$\text{foam capacity } c_F = 1 - \frac{V_{foam}}{V_{free}} \quad (3)$$

Depending on the investigated foamable mixture, gas load factors of $f_G = 0.21 \dots 0.25 \sqrt{Pa}$ were achieved. A foamable mixture of chlorobenzene/ethylbenzene (CB/EB) (sample mass: 1 kg) with impurities of iron oxide particles was used. In preliminary investigations, this mixture showed constant foaming and thermal stability. An equimolar composition of $0.5 \text{ mol}_{CB} \cdot \text{mol}_{total}^{-1}$ with a liquid density of $0.9756 \text{ g} \cdot \text{cm}^{-3}$ was used for distillation. The compositions of the bottom and head products were determined by multiple measurements. All density measurements are carried out at 20°C with the density meter DMA 4100 M (Anton Paar GmbH, Graz/Austria). The composition was determined using a calibration function [17] of the mixture densities of CB/EB. To determine the separation performance of the packings, the Fenske equation [18] with the separation factor $\alpha_{12} = 1.1019$ at 1 bar abs was applied to calculate the minimum number of theoretical stages.

The foamable mixture was processed in combination with three different packing types. Besides the structured packing M350.Y (Sulzer Chemtech AG, Winterthur/Switzerland), random packings of Raflux rings (RVT Process Equipment GmbH, Marktrodach/Germany) in two different material versions were tested.

Table 1. Properties of the used packing internals.

packing characteristics	unit	M350.Y 	Raflux ring 	Raflux ring 
material	-	stainless steel	Perfluoroalkoxy polymers (PFA)	stainless steel
size per packing element (height h x diameter d)	mm	100 x 45	15 x 17	15 x 17
number	-	5	181	181
total weight	kg	0.18	0.16	0.29
total packing height	m	0.54		
spec. surface a	m^2/m^3	350	240	302
bulk density in column	kg/m^3	165	152	271
inner oil heated column surface	m^2	0.09		
packing surface	m^2	0.35	0.25	0.32
separation surface (packing + column)	m^2	0.44	0.34	0.41
void fraction	%	99	91	96

Distillation throughput and separation performance

The liquid load w_L as a function of gas load factor f_G is shown in Fig. 2. It illustrates the reduction of the distillation throughput due to foam within the packings. The comparison of w_L without packings (no packings) with and without foam illustrates that foam itself does not lead to a reduction in liquid load and thus in throughput. The pressure drops caused by packings led to throughput losses of 19 % on average. The largest losses of 30 % in the distillate stream occurred due to foam in the structured packings. Obviously, any increase in pressure drop, due to packings and especially due to foam in packings, adversely affects the fluid dynamic with reduced throughput and distillate flows as a consequence.

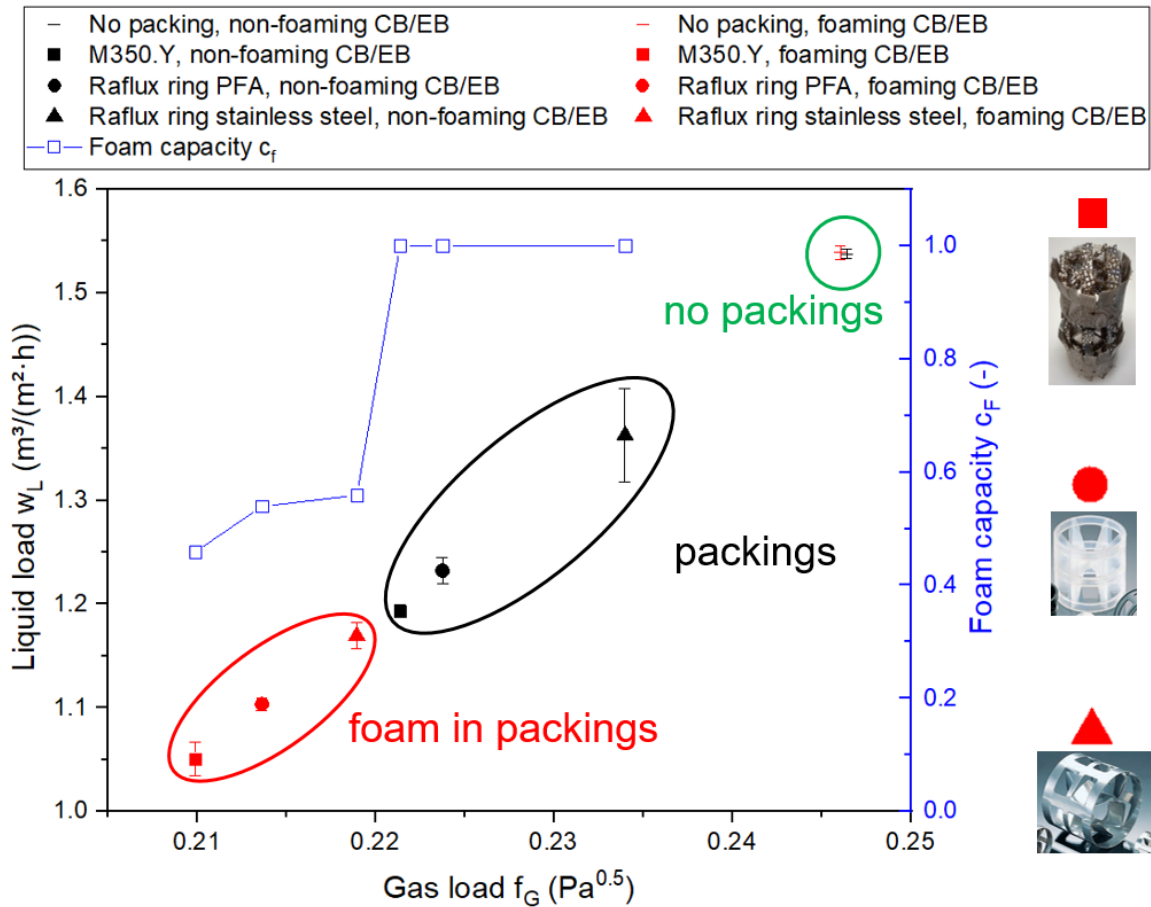


Fig. 2. Liquid load w_L and foam capacity c_F as a function of the gas load f_G in distillations.

The most common parameter for foam detection in industrial operation of distillation and rectification plants is the differential pressure. The length-related pressure drop of foam-filled packing increased by 13 mbar/m for the structured packing M350.Y, 25 mbar/m for the Raflux PFA packing and 17 mbar/m for Raflux stainless steel compared to foam-free operation. The pressure drop caused by foam is avoidable and occurs additionally to the pressure drop caused by the internals themselves. These foam-induced pressure drops are high and usually lead to build-up of liquid and entrainment. Consequently, a reduction in separation performance occurs.

Fig. 3 presents the influence of foam on the separation performance of the three investigated packings. The separation performance is quantified by the Height Equivalent to one Theoretical Plate (HETP) and its reciprocal, the Number of Theoretical Stages per Meter (NTSM). It must be mentioned that the inner surface of the heated column accounts for 21 % (M350.Y) ... 36 % (PVA Raflux Ring) of the total separation surface. This constant influence of the heated double jacket column led to increased separation efficiencies, which resulted in relatively low HETP values. The structured packing M350.Y achieved the highest separation performance with the lowest HETP values with and without foam loading. However, the structured packing is also the most susceptible packing to foam. Foam rose fastest and highest in the regularly shaped channels of the packing. Therefore, its separation performance decreased the most under foaming conditions. The random stainless steel Raflux packing showed the lowest decrease of separation performance as a result of foam. Overall, Raflux rings made of perfluoroalkoxy polymer (PFA) showed the lowest separation performance with the highest measured HETP values. This may be attributed to the material and surface properties of the polymer, which lead to a lower wetting effect, compared to steel. In general, it is noticeable that the investigations were carried out at relatively low gas loading factors of $f_G = 0.21 \dots 0.25 \sqrt{Pa}$. However, it is expected that both the quantitative effect of foam on the separation efficiency of the various internals and their relative susceptibility to foam can also be observed under technically relevant process parameters.

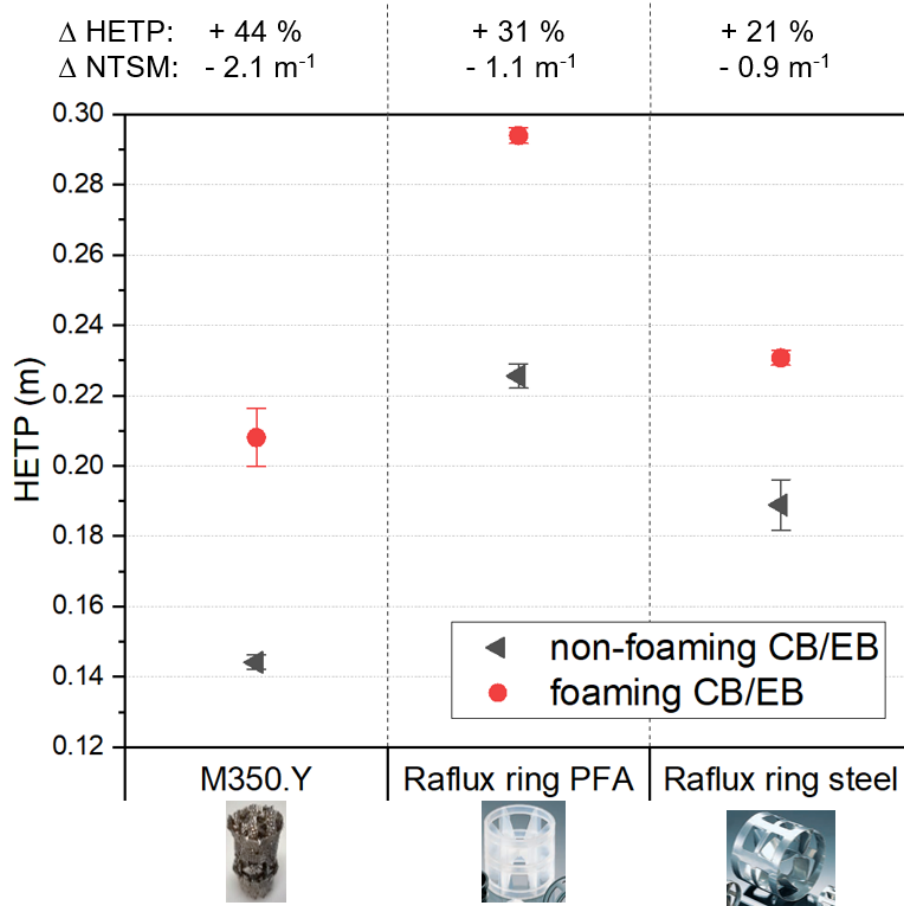


Fig. 3: Reduction of separation performance due to foam in packings at $f_G = 0.21 \dots 0.25 \sqrt{Pa}$.

In order to quantify the influence of foam on the distillation in packed columns, the results from Fig. 2 und Fig. 3 with and without foam loading can be interpreted as follows. Without foam loading and a foam capacity of $c_F = 1$, the 25 % higher separation performance of M350.Y is offset by a 13 % lower throughput compared to the Raflux stainless steel rings. In comparison to the randomized PFA rings, a 36 % higher separation performance was measured using the structured packings M350.Y, while the throughput was 6 % lower. With foam loading, i.e. at a reduced foam capacity $c_F < 1$, a 9 % and 28 % higher separation efficiency was measured for the M350.Y structured packing compared to the Raflux stainless steel and PFA rings, respectively. The structured packing destabilized the foam less effectively. This resulted in a 10 % higher reduction in throughput and foam capacity under foam loading compared to the stainless steel version of the Raflux rings.

These results are related to the lower transverse mixing of the fluid in the perforated M350.Y packing. Greater transverse mixing and qualitatively more turbulent fluid dynamics were observed within the random packings, destabilizing the foam more effectively.

Conclusions and Outlook

This study of evaporation-induced foam in structured and random packings in a laboratory DN50 distillation setup provides first insights to the effects of foam on distillation throughput and separation performance. The lab scale distillation column was operated under total reflux, following a standard procedure for separation efficiency and fluid dynamic capacity measurements.

Under consideration of the linear relationship between gas loading and liquid loading as a measure of distillation throughput, it was shown that foam in packings reduces gas loading and thus distillation throughput equally. More pronounced turbulent fluid dynamics in the random packings destabilized the foam more effectively, than perforated

structured packings. Foam interferes with mass transfer and thus reduces separation efficiency. In absolute terms, foam-loaded structured packings showed the highest separation performance compared to the randomized packings.

In future studies foamable mixtures based on temperature resistance surfactants as surface-active component will be investigated as a model system with constant foamability. Furthermore, in terms of transferability between different foamable mixtures, the use of aqueous solvent mixtures is conceivable.

Acknowledgements

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