

# Distillation of Systems Containing Two Liquid Phases

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## Introduction

It is sometimes found that a distillation column in which two liquid phases appear must be operated at a lower vapor rate than a column for a similar system of one liquid phase. The two-liquid-phase system is believed to foam and a derating factor, known as a foaming factor, is used to design the column. No previous work known to us has reported observations of foaming in distillation columns with two liquid phases. Most of the previous work on foaming in distillation has investigated the Marangoni effect and has shown that more foam is observed in a column where the liquid surface tension is lower at the top of the column than at the bottom (positive system) than where the surface ten-

sion gradient is reversed (negative system) (Bainbridge and Sawistowski, 1964; Fane and Sawistowski, 1969; Zuiderweg and Harmens, 1958).

Ross and Nishioka (1975, 1981) investigated the foaminess, on a single plate, of a liquid mixture that was caused to change from a single phase to two liquid phases by blowing gas through the liquid, which changed the composition of the liquid by preferential evaporation. The foam height increased as the transition from one to two liquid phases was approached. However, the maximum foam height occurred at the point of transition, and then reduced when two liquid phases were formed. It was concluded that one of the separated liquids acted as a defoamer

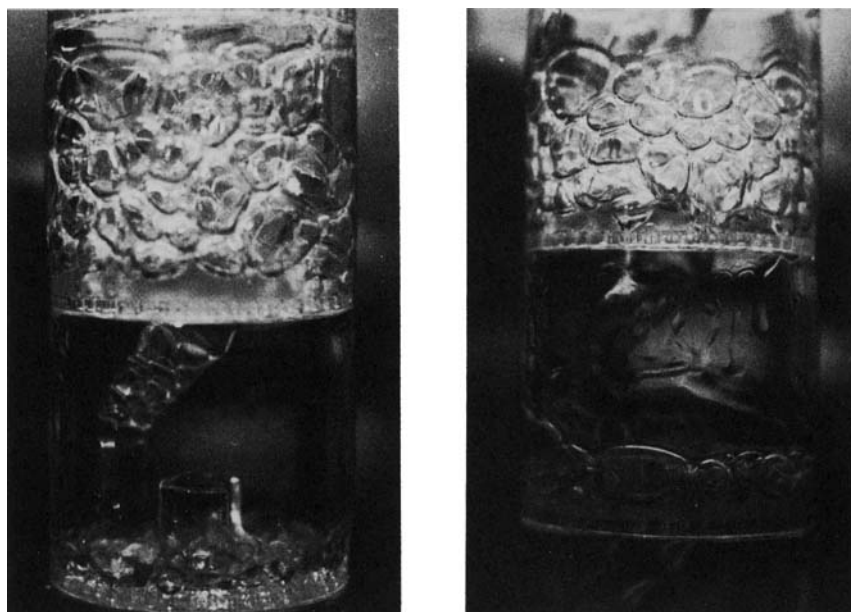
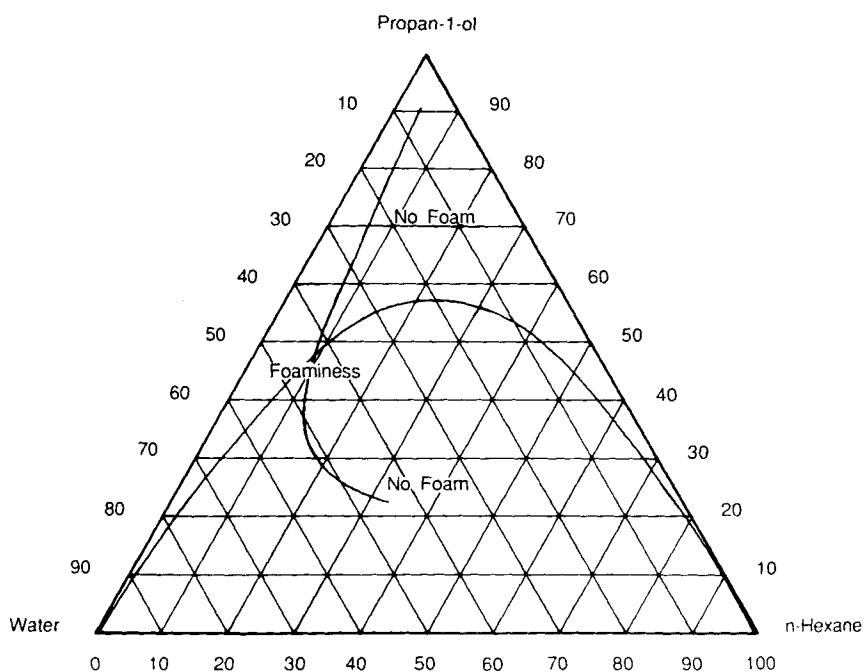


Figure 1a. *n*-hexane-propan-1-ol-water system in 30 mm dia. Oldershaw column.  
(a) Single to two-liquid phase transition, bottom column section.



**Figure 1b, c.** *n*-hexane-propan-1-ol-water system in 30 mm dia. Oldershaw column.  
 (b) Whole column.  
 (c) Two-liquid phase, top column section.



**Figure 2.** *n*-hexane-propan-1-ol-water at 100°F.  
 All concentrations in wt. %.

for its foaming conjugate. Within the two-liquid-phase region foam is inhibited. This phenomenon has not been previously reported in actual distillation columns although it is reasonable to expect that similar phenomena will occur.

### Experimental Work

A 50-plate 30 mm dia. Oldershaw distillation column was used in the present study to examine the behavior of systems chosen so that a transition from one liquid phase to two liquid phases could occur. The following ternary systems were used:

1. *n*-hexane-propan-1-ol-water
2. Cyclohexane-propan-1-ol-water
3. *n*-hexane-ethanol-water
4. Methyl ethyl ketone (MEK)-methanol-water

For systems 1 to 3 a single liquid phase was present on the plates at the bottom of the column and two liquid phases were observed on the plates at the top. For system 4 the single liquid phase was at the top and two liquid phases at the bottom.

### Observations

In all of these systems it was observed that the plates in the top and bottom sections of the column (approximately 15 plates in each section) did not foam, while the plates in the middle section (approximately 20) exhibited significant foaming which completely filled the tray spacing; these conditions are shown in Figure 1. During the experiments a hot-air jacket was placed around the outside of the column to prevent heat losses; the jacket was removed for greater clarity in the photographs.

Figure 2 shows a typical distillation line for one of the experiments (*n*-hexane-propan-1-ol-water) passing through the one-liquid-phase/two-liquid-phase boundary.

It was also observed that the number of plates in the middle of the column that were foaming depended on the composition of the original mixture charged to the still. If the original mixture was *n*-hexane rich, then more plates foamed than if the mixture was water-rich.

It thus appears that the capacity of columns in which there is a transition from one liquid phase to two liquid phases (or vice versa) will indeed be limited by foaming, but that the foaming occurs only in those plates with a liquid composition near that of the one-phase/two-phase transition.

### Literature cited

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