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Intermittent feeding batch distillation with liquid exchange between two columns for separation of binary mixtures

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ABSTRACT

Batch distillation column with top vessel and bottom vessel is a new and promising structure. In the basis of it, a new operation strategy that two columns operate in parallel with liquid exchange under total reflux is proposed to separate binary mixtures in this article. A mathematical model is setup for the simulation. Operation time and yield of different feeding concentration and holdup in vessels are compared and discussed. The results show that there is an optimal holdup for different feeding concentration and the total yield increases with the growth of total charge. Furthermore, the proposed operation mode is verified experimentally and it reveals significant potential for separation with higher efficiency and lower pressure drop.

1. Introduction

Batch distillation is widely used in the fine and specialty chemical and pharmaceutical industries for purifying or recovering high-value-added low-volume products because of its advantages such as flexibility and simplicity of operation, little capital investment, multiple products with a single column and so on. With an increasing demand for high quality product, Some new operating modes in batch distillation process have been proposed and studied to improve separation efficiency or flexibility in the past two decades, such as total reflux operation [1–3], middle vessel operation [4–10], multivessel operation [11–13], multi-effect batch distillation [14] and so on.

The total reflux operation is one of the promising modes of batch distillation. The most common cycling control proposed by Perry is that the column is operated at infinite reflux ratio and the light component is accumulated in the reflux vessel as the process proceeds [15]. The column proceeds the total reflux operation until the concentration in the reflux drum reaches the specified value. The holdup in reflux vessel is drained away as the top product. And then the column returns to the total reflux operation again. If another reflux vessel is added to be used alternatively, the time of recovering the product and filling vessel is saved [16]. In some cases, the purpose of the distillation is to recover both components of a binary mixture at a very high degree of purity. The first method is proposed by Treybal to solve the problem with batch distillation [17].

For separating small relative volatility mixture, the number of stages is pretty large. Connection between the columns is an effective way to make sure distillation column is not too high. Conventional connection system is that a high column divides into several short columns. The total stages are the addition of all columns. However, it also causes that the total pressure drop is added. Gas and liquid mass transfer happens in each column in turn during start-up. It needs a lot of time to realize equilibrium. And the system is fragile. If one column of the system fails, the separation needs to start again.

In this article, a new operation strategy of liquid exchange between two columns is proposed to separate binary mixtures and a mathematical model for the simulation is set up. The simulation results are analyzed and some of the parameters are optimized preliminarily. The new operation strategy is applied for the separation of mixtures with different initial concentrations of feed in laboratory-scale distillation columns.

2. New operation strategy

The structure of intermittent feeding batch distillation column is shown in Fig. 1. There are two top vessels and two bottom vessels added to a batch distillation column. At the beginning, a certain amount of feed is added to the column, one top vessel, one bottom vessel and reboiler. The column is operated with total reflux. Once the product (in top or bottom vessel) reaches the specified purity, the vessel containing the product is isolated from the column and

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Nomenclature

α	Relative volatility
F	Initial feed (mol)
F_T	Total feed (mol)
H	Holdup in the top vessel (mol)
H_i	Holdup on the *i*th stage (mol)
H	Holdup in the reboiler and bottom vessel (mol)
H	Holdup in the reboiler (mol)
i	Theoretical stage
j	Component
L	Liquid flow (mol/min)
L_i	Liquid flow leaving the *i*th stage (mol/min)
n	Number of stages without vessels
N	Total number of theoretical stages
V	Vapor flow (mol/min)
V_i	Vapor flow leaving the *i*th stage (mol/min)
x_{Ab}	Concentration of the light component in the bottom vessel of column A
x	Concentration of the light component in the top vessel of column A
x_{Bb}	Concentration of the light component in the bottom vessel of column B
x_{Bt}	Concentration of the light component in the top vessel of column B
x_F	Initial feed composition
$x_{i,j}$	Liquid concentration of the *j*th component leaving the *i*th stage, mole fraction
x_p	Product purity
$y_{i,j}$	Vapor concentration of the *j*th component leaving the *i*th stage, mole fraction

the product is collected from the vessel. At the same moment, the other vessel which has already been charged with the same amount of fresh feed as the product is connected to the column. The operation is achieved by switching the valves. The two vessels are used alternatively. The advantages of the intermittent feeding batch distillation column is that the capacity of the column is more flexible. Different amount of feed can be separated by the same column without start-up many times. The product of both the light component and the heavy component can be collected before the separation is completed.

Based on the structure of intermittent feeding batch distillation column, A promising new operation mode is proposed. Liquid in the vessels is exchanged with the liquid in other columns. There are some differences in the case of multiple columns. After total reflux operation, liquid in the vessel (connected to the column) is transferred to the free vessel (unconnected to the column) of other column or collected as the product. The free vessel (unconnected to the column) is used to receive the liquid from other column or add fresh feed. The purpose of liquid exchange is to obtain purer product, especially binary mixtures with small relative volatility like isotope. The effect of liquid exchange is similar to the connection of columns. But the method of liquid exchange is better than the conventional connection. Because the columns run independently in most of the time. If one of the columns breaks down, other columns are unaffected. And there is no superposed pressure drop, which is helpful to separate heat-sensitive mixtures. This article presents the results of some initial exploration of the liquid exchange between two columns. The method of liquid exchange in multiple columns is being studied and the results will be presented in future articles.

The whole operation contains start-up, recovery of the light component from the top vessel of the right column and recovery of the heavy component from the bottom vessel of the left column. Liquid in the top vessel of left column and bottom vessel of right column should be exchanged to set up the concentration gradient in the middle period of start-up as the Fig. 2. To avoid backmixing while liquid exchange and charging fresh feed, the concentration of holdup between two vessels or between a vessel and fresh feed should be close. The light component in the left column should be transferred to the right column and the heavy component in the right column should be transferred to the left column. All above, three kinds of operation strategies are proposed to meet the conditions of feed with different concentration (the following low, intermediate and high concentration refers to the concentration of the more volatile component).

2.1. Low concentration strategy

After the concentration gradient of two columns is set up, the concentration of the light component in different vessels can be considered as follows: bottom of left column < bottom of right column < feed < top of left column < top of right column.

In the case of low concentration feeding, liquid concentration in vessel 1 is most close to feeding. So vessel 1 can be used as feeding

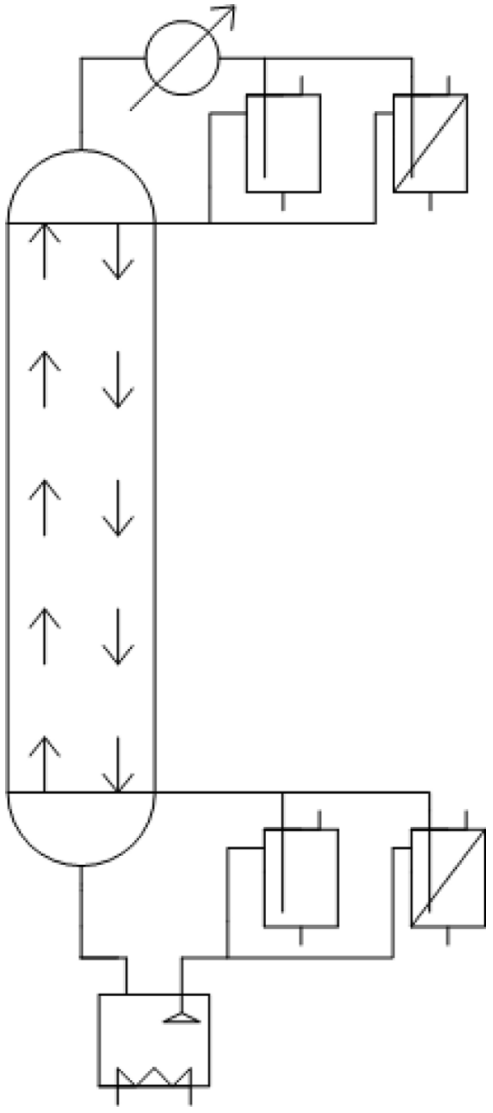

Fig. 1. The structure of intermittent feeding batch distillation column.