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A new application of dividing-wall columns for the separation of middle-boiling impurities

*Luca F. Cameretti**, *Daniel Demicoli*, *Ralf Meier*

Evonik Industries AG, Paul-Baumann-Str. 1, 45772 Marl, Germany

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ABSTRACT

There is growing interest in dividing-wall column (DWC) technology, and its simplicity lies at the heart of its appeal. Compared to conventional multicolumn arrangements a DWC can generate capital cost savings of up to 40% simply because less equipment is required. With their greater thermodynamic efficiency, dividing-wall columns can also reduce operating costs by around 30% (Asprion and Kaibel, 2010). The actual savings that can be realized will depend on the specific application.

In general, the use of DWCs is recommended for multicomponent liquid mixtures that are to be separated into at least three fractions with high purity requirements. Further, DWCs are especially favorable for the separation of small quantities of light and heavy boilers from the main middle-boiling product (Dejanović et al., 2010; Niggemann et al., 2010).

In this paper, a new configuration of divided wall column is presented, which is most effective for the separation of a feed containing low concentrations of middle-boiler (about 1% or less) into a pure distillate and a pure bottoms fraction, while the impurity is concentrated in the side-stream. Product loss in the side-stream is a major cost-driver. In the DWC-configuration proposed here, product loss can be cut in half compared to a single side-stream column.

Experiments at pilot scale have confirmed the simulation results, proving a new opportunity to profit from the vertical wall inside the distillation column.

1. Introduction

Albeit the fact that distillation is one of the most ancient techniques to separate multicomponent mixtures, research is still done in order to tease out the last percentages of cost saving potential. One big step was surely achieved by better heat management. Although the idea of heat coupling is known at least since the 1930s, Petlyuk et al. (1965) widely introduced the separation of a three-component system in a coupled setup of prefractionator and main column in the mid 1960s (Dejanović et al., 2010). Subsequent development by Kaibel (1987) has led to the first technically used dividing-wall columns (DWC), where prefractionator and main column are combined in one shell.

The principle and many variations of DWCs have already been dealt with in literature. Dejanović et al. (2010) and Yildirim et al. (2011) give excellent reviews on state-of-the-art and current activities. They and the cited authors show different setups containing one or more dividing walls positioned at the top, the bottom, or in the middle of the column in order to achieve pure products out of a multicomponent feed. All dividing-wall columns shown in the literature have at least one common packed section, i.e. a section without dividing wall. Further, in most of the examples given, the middle-boiling fraction predominates the feed composition.

In the present case, a pseudo three-component feed containing less than 1% of middle-boiler has to be separated into pure top and pure bottom products. The middle-boiler has to

* *Corresponding author*. Tel.: +49 2365496722.
E-mail address: luca.cameretti@evonik.com (L.F. Cameretti).
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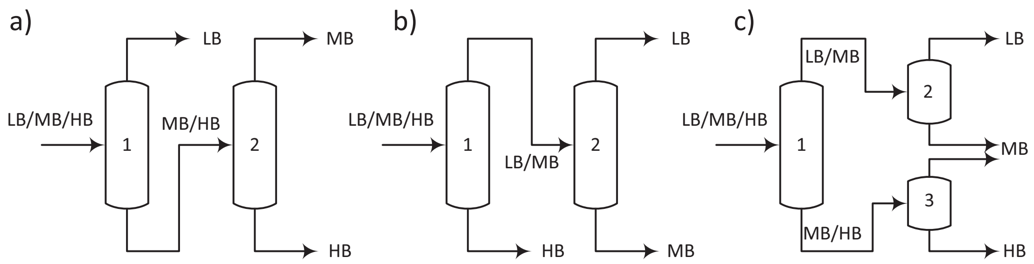

Fig. 1 – (a) Direct, (b) indirect, and (c) sloppy distillation sequence for the separation of a three-component mixture LB/MB/HB.

be concentrated in the side-stream, which will then be incinerated. Loss of low and high boiler in the waste stream is a major cost-driver. A further challenge lies in the fact, that top product and heavy-boiler are narrow boiling ($\alpha \sim 1.4$–2.4). Furthermore, the vapor pressure curves of light-boiler and middle-boiler are very close to each other ($\Delta T < 5$ K); the phase equilibrium also reveals an azeotrope.

2. Methodology

The following sections show the steps to the final design of the dividing-wall column suitable for the separation of small quantities of middle-boiler from a narrow-boiling mixture. Several column setups were simulated with the commercial simulator ASPEN Plus® and benchmarked against each other. In order to facilitate reading, following abbreviations will be used: light-boiler (LB), middle-boiler (MB), and high-boiler (HB), respectively.

2.1. *The classical separation sequence*

There are three classical separation sequences of simple columns for a mixture of three components: the direct, the indirect, and the sloppy configuration, as shown in Fig. 1.

Due to the prevailing phase equilibria mentioned above (low relative volatility and azeotropic behavior between LB and MB), the indirect path appears to be more suitable at first sight. The theoretical number of stages for column 1 and 2 were estimated using the DSTWU model (Gilliland, 1940; Underwood, 1932; Winn, 1958). The typical correlation between reflux ratio and number of stages for different recovery rates of LB are shown in Fig. 2. The plots make clear that, a very high number of theoretical stages is required, especially for the separation of LB and MB in the second column, leading to high investment (size of the equipment) and operating expenses (reboiler and condenser duties). Another drawback is the loss of valuable LB in the MB fraction. Hence, a classical separation sequence is economically not feasible.

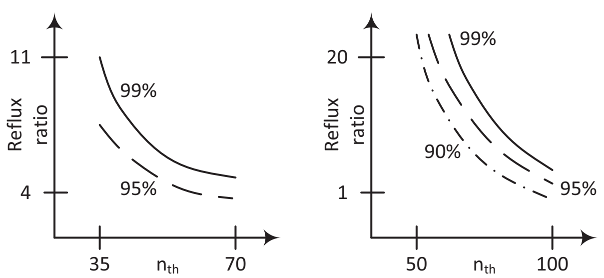

Fig. 2 – Reflux ratio vs. theoretical number of stages for (a) LB/HB separation with a recovery of 99% and 95% of LB and 0.1% of HB in the distillate; (b) in analogy for the separation of LB/MB.

2.2. *Side-stream columns (SSC)*

The next evolution step is to use side-stream columns. The obvious advantage of a SSC is that it combines two columns in one shell, hence reducing investment costs. In the present case, however, a single SSC will require extremely high reflux ratios in order to concentrate the MB up to a sufficiently high fraction in the side-stream. Hence, a SSC configuration will lead to either large equipment dimensions and high energy cost or high product loss.

At first sight, a setup consisting of a side-stream stripper or rectifier column attached to the main tower appears worth consideration. However, they are not applicable when the side-stream withdrawn from the main column contains both LB and HB in considerable concentrations.

A combination of two SSCs can overcome this constraint. In order to minimize loss of LB as well as HB and at the same time decrease energy consumption, LB and HB are separated as top and bottom products in a first SSC, while MB is concentrated inside the column. This is done at moderate reflux ratios, high enough to obtain pure LB but well below the value required to achieve the desired MB concentration in the side-stream. The resulting MB peak is tapped by a side-stream which then will still contain a high amount of valuable LB and HB. This side-stream is fed to a second SSC which basically has the same separation task as the first one: it separates pure LB as top product, concentrates the MB in the side-stream, and returns HB without LB. The advantage, though, is that MB is already concentrated to some extent in this stream. Since the flowrate is much lower than for the first column, very high reflux ratios and hence higher MB concentrations can be achieved at low cost.

The column arrangement is shown in Fig. 3a.

2.3. *Dividing-wall column (DWC)*

The obtained combination of two SSCs can be further optimized, especially concerning investment costs. Since both columns produce high-purity products LB and HB, they can share just one condenser and one reboiler, respectively. In addition, they can be combined in a common shell. This leads to a dividing-wall column with a dividing wall extending over the whole active height from the bottom to the top stage. The withdrawal from the first to the second side-stream column is then designed as a pump-around from the left-hand side (LHS) to the right-hand side (RHS) of the DWC. The setup is schematically shown in Fig. 3b. While the product compositions and energy demand of two coupled SSCs and the DWC are identical, investment for the DWC will obviously be significantly lower.