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Vapour- and liquid-side volumetric mass transfer coefficients measured in distillation column. Comparison with data calculated from absorption correlations

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Abstract

Volumetric mass transfer coefficients in liquid and vapour phases in distillation column were measured by the method consisting of a fitting of the concentration profile of liquid phase along the column obtained by the integration of a differential model to the experimental one. The mathematical model of distillation process includes mass and energy balances and the heat and mass transfer equations. The film model flux expressions with the convective transport contributions have been considered in the transfer equations. Vapour and liquid phases are supposed to be at their saturated temperatures along the column. Effect of changes of phase flows and physical properties of phases on the mass transfer coefficients along the column and non-ideal thermodynamic behaviour of the liquid phase have been taken into account. The concentration profiles of liquid phase are measured in the binary distillation of the ethanol–water and methanol–ethanol systems at total reflux on metal Pall Rings and Intalox saddles 25 mm in the column with diameter of 150 mm. The distillation mass transfer coefficients obtained by the fitting procedure are compared with those calculated from absorption data using Onda's, Billet's and Linek's correlations. The distillation heat transfer coefficients calculated from the model assuming saturated temperatures in both phases are compared with those calculated from the Chilton–Colburn and penetration model analogy between mass and heat transfer. The results have confirmed an agreement neither between distillation and from absorption correlations calculated mass transfer coefficients nor between analogy and from enthalpy balance calculated heat transfer coefficients. Also the concentration profiles obtained by the integration of the differential model of the distillation column using the coefficients from absorption correlation have differed from the experimental profiles considerably.

Keywords: Mass transfer; *HETP*; Distillation; Packed column; Absorption

1. Introduction

Distillation columns are the most widely used large-scale separation equipments in chemical, food and pharmaceutical industry. Energy demands of the distillation are extremely high and its recovery for further purposes is difficult, therefore the capital and operational costs of the distillation process are high. Even though alternative separation techniques are still developed, they cannot replace the distillation process completely, what makes its future use indubitable. An attention hence should be paid to the distillation as a small improvement in its design or performance has a large financial impact. Despite the earnest effort of many chemical engineers and a huge number of original works submitted up to present time, the design of distillation columns has long been relying on experience and is essentially empirical in nature.

The idea of the height equivalent to a theoretical plate is most commonly used to the column design. An integral character of *HETP* makes it impossible to find the universal rule for *HETP* data transfer among different systems and conditions. Another approach employs the fact, that absorption and distillation have the same physical background—interfacial mass transfer between gas and liquid phase which is characterised by mass transfer coefficients in liquid $k_L a$ and gas $k_V a$ phase. An effort to calculate the *HETP* using absorption mass transfer coefficients recalculated to the conditions in distillation column has

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mostly led to large deviations from the experimental data (often higher than 50%). The discrepancy can be ascribed partially to large extrapolation in temperature used in the data transfer from absorption to distillation. However, the measurements of mass transfer coefficients directly in distillation columns are rare (Kayihan et al., 1977; Honorat and Sandall, 1978; Dribika and Sandall, 1979). In distillation namely, it is not possible to measure individual mass transfer coefficients by using a proper distillation system in which the overall resistance to the interfacial mass transfer is mainly due to one of the phases and to study behaviour of the individual coefficients separately as it is possible in absorption. An assumption that all of the resistance to mass transfer is in the vapour phase has been used in the mentioned papers in which the mass transfer coefficients in distillation columns were measured. Arwikar and Sandall (1980) have shown, however, that this assumption is mistaken and thereby they infirm the principal conclusion of the previous works, namely that the mass transfer coefficients calculated from absorption correlations (to be specific the correlation by Onda et al., 1968) fit well the coefficients in distillation columns.

Recently, Linek et al. (2005) presented a method of simultaneous determination of volumetric mass transfer coefficients in both phases in distillation column. The method consists of a fitting of the concentration profile along the column obtained by the integration of a differential model to the experimental one. The importance of the calculation methods based on the integration of differential equations describing a distillation column has increased. The methods, designed as "rate-based" or "non-equilibrium", require thorough understanding of the processes occurring inside a distillation column as they utilize a numerical solution of energy and mass balances with transport equations describing components, phases and energy flows within the column, covering all the process determining phenomena (Mori et al., 1996, 1999, 2002, 2006; Taylor and Krishna, 1993; Wesselingh, 1997). The lack of available and reliable values of the coefficients, which characterise rates of the phenomena included in the rate-based models, has braked the development of more accurate and reliable models for the design of distillation columns. The use of the mass transfer coefficients determined under the conditions of distillation columns would significantly improve the reliability of the simulation models.

Practicability of the method proposed by Linek et al. (2005) was preliminary verified on the limited data set (Pall-ring 25, ethanol–water (E–W), reboiler duty 25 kW). The E–W distillation system was used for sharp changes of the equilibrium coefficient with concentration (i.e., along the column), which makes it possible to separate the individual phase contributions to interfacial mass transfer resistance and thus to evaluate mass transfer coefficients in both phases simultaneously.

The aim of this work is to apply this method on wider set of data, i.e., on another type of packing (Intalox-25) and distillation system (methanol–ethanol (M–E)). The distillation system M–E shows small changes of the equilibrium coefficient with concentration in comparison with the originally used E–W system. The distillation mass transfer parameters measured by this method are compared with those calculated from absorption correlations.

2. Theoretical

2.1. Differential model of distillation column

Linek et al. (2005) presented the model with some errors. This is why we present it here again. The model is based on the application of mass and energy balances and heat and mass transfer equations to a differential control volume $S\,\mathrm{d}z$ of the column (see Fig. 1). The balances and transfer equations have been developed under following assumptions: (i) distillation of binary mixture under total reflux; (ii) process is adiabatic and steady state; (iii) the overall pressure is constant through out the column; (iv) the plug flow in both phases; (v) interfacial mass and heat transport are described using a film model in which the convective flow is taken into account; (vi) the rate of heat transfer is such as to keep the vapour and liquid phases just at their dew and boiling points respectively; (vii) interfacial area for the mass and heat transfer is identical.

Global mass, component and enthalpy balances simplify under the total reflux and adiabatic conditions to

$$L - V = 0, \tag{1}$$

$$x_A - y_A = 0, \tag{2}$$

$$L\left(\frac{\mathrm{d}h_L}{\mathrm{d}z} - \frac{\mathrm{d}h_V}{\mathrm{d}z}\right) + \frac{\mathrm{d}L}{\mathrm{d}z}(h_L - h_V) = 0. \tag{3}$$

Liquid phase mass balances of components A and B (the molar fluxes $N_{A,B}$ are defined to be positive from the vapour to the liquid) are

$$L\frac{\mathrm{d}x_A}{\mathrm{d}z} + x_A\frac{\mathrm{d}L}{\mathrm{d}z} - N_A = 0,$$

$$L\frac{\mathrm{d}x_B}{\mathrm{d}z} + x_B\frac{\mathrm{d}L}{\mathrm{d}z} - N_B = 0. \tag{4}$$

$N_{A,B}$ are given by following rate and equilibrium relations and by the mass continuity requirement at the interface (an equal

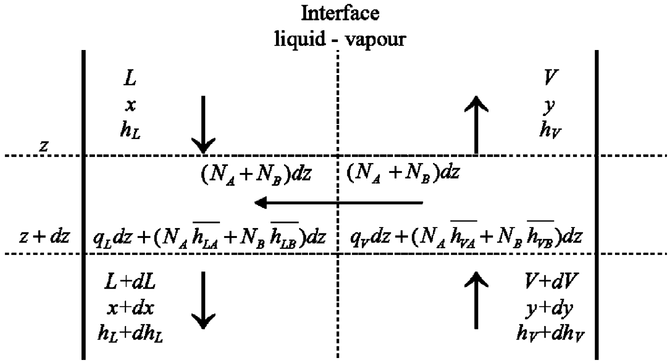

Fig. 1. Differential control volume of distillation column.