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Geometry and viscosity effects on separation efficiency in distillation

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ABSTRACT

In a distillation process the physical properties of the mixture, operating conditions and the geometry of the packing determine fluid dynamics and mass transfer and thereby the performance of the column. Liquid viscosity has an impact on separation efficiency as well as on capacity. Measurements were carried out with two test mixtures, CB/EB as reference system and one with elevated viscosity, using two different structured packings. Based on experimental data, different predictive models were assessed with respect to their capability to describe the impact of viscosity on column efficiency.

Increase of viscosity by a factor up to 6 resulted in a decrease of separation efficiency by 50%, higher pressure drop and lower capacity. The measurement of liquid hold-up revealed no changes due to packing geometry, but a significant increase for higher viscosities. Whilst separation efficiency at low viscosity was sufficiently met by the rate based models, the influence of increased viscosity could not be predicted correctly.

The results reveal that even at moderate viscosities column performance is affected significantly. Hence, if rate based models are applied to conditions deviating from those they were validated for, their accuracy has to be considered carefully.

1. Introduction

Structured packings are widely used in distillation and absorption processes, since they combine high capacity and good separation efficiency. These are determined by the geometry of the packing, operating conditions and the physical properties of the mixture. Significant effort has been made to find and improve predictive models for structured packings. Thereby, the calculation of efficiency in dependence from geometry and physical properties is desired to avoid time consuming and expensive experiments. However, experiments are needed for model validation whenever major changes in physical properties or geometry are made. In this respect, liquid viscosity is especially important, since it has an impact on separation efficiency as well as on capacity, because it influences fluid dynamics and mass transfer in the liquid phase. Typically, efficiency tests are carried out using standard test mixtures, based on the comprehensive compilation of Onken and Arlt (1990). Most frequent systems are Chlorobenzene/Ethylbenzene (CB/EB) and Cyclohexane/n-heptane (C6/C7). Increased viscosities are represented by Trans-/Cis-Decalin, however well below 1.5 mPa s at distillation conditions. There is little data available in open literature concerning the influence of viscosity beyond this value. Hence, datasets used for the validation of existing rate based models are limited to the low viscous mixtures stated above.

Böcker and Ronge (2005) used polymers in distillation experiments to increase viscosity and study its impact. They found a reduction of separation efficiency by 50% at $\mu_L = 25$ mPa s and by 80% at $\mu_L = 260$ mPa s. Since the polymer could not be vaporized, a stripper operation was used instead

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Nomenclature

a_p	[m^2/m^3] nominal specific surface area of the packing
$a_{c,av}$	[m^2/m^3] specific surface area averaged over column cross section
a_{eff}	[m^2/m^3] effective (interfacial) area
A_{pe}	[m^2] surface contributed by packing element
A_{wall}	[m^2] surface contributed by column in height of packing element
A_{wipers}	[m^2] surface contributed by wall wipers of one packing element
b	[m] corrugation base length
d_h	[m] hydraulic diameter of flow channel, $d_h = (2bh)/(b+2s)$
d_p	[m] diameter of packing element
F_m	[$Pa^{0.5}$] vapor load averaged over column height
g	[m/s^2] gravitational acceleration
h	[m] corrugation height
h_L	[$m^3{}_L/m^3{}_p$] liquid hold-up
$h_{L,\ load}$	[$m^3{}_L/m^3{}_p$] calculated hold-up in loading region
$h_{L,\ preload}$	[$m^3{}_L/m^3{}_p$] calculated hold-up in preloading region
$h_{L,\ preload,\ eff}$	[$m^3{}_L/m^3{}_p$] hold-up in preloading region calculated with a_{eff} and $\delta_{L,\ eff}$
h_p	[m] height of packing element
HTU_L	[m] height of liquid mass-transfer-unit
HTU_{Vo}	[m] height of overall vapor phase related mass-transfer-unit
HTU_V	[m] height of vapor mass-transfer-unit
k_V	[m/s] vapor-side mass-transfer coefficient
k_L	[m/s] liquid-side mass-transfer coefficient
M	[mN/m] stabilizing index
$\Delta p/\Delta z$	[Pa/m] specific pressure drop per packing height
s	[m] corrugation side length
u_{Ls}	[m/s] superficial liquid viscosity
$u_{V,eff}$	[m/s] effective vapor velocity
$w_{L,m}$	[$m^3/(m^2\ h)$] liquid load averaged over column height
x_1	[mol_1/mol_{tot}] liquid phase mole fraction
$x_1{}^*$	[mol_1/mol_{tot}] liquid phase mole fraction of condensate in equilibrium with x_1

Greek letters

α	[°] corrugation inclination angle
α_L	[°] effective flow angle of liquid on packing surface
α_{12}	[dimensionless] separation factor
δ_L	[m] liquid film thickness
$\delta_{L,\ eff}$	[m] liquid film thickness, calculated with effective line load
ε	[dimensionless] void fraction of packing
λ	[dimensionless] stripping factor
μ_L	[Pa s] liquid viscosity
ρ_L	[kg/m^3] liquid density
σ	[N/m] surface tension

of distillation under total reflux. Unfortunately, addition of a non-volatile component adds a mass transfer resistance at the vapor/liquid interface due to the accumulation of the latter to the viscosity effect. Furthermore, due to the application of a polymer, the local viscosity instead of bulk viscosity might be relevant for mass-transfer (Rondelez et al., 1987; Tsai et al., 2009).

For sieve trays, viscosity was found to have a strong impact below 30 mPa s, causing tray efficiency to decline about 50% depending from operational and geometric conditions (Mahiout and Vogelpohl, 1987). In connection to the need of efficient solutions for CO_2 absorption, recent studies focussed on the improvement of mass-transfer for high viscosities. This demand arises from the increase of viscosity if the water content of aqueous amine solutions is reduced. Therefore, macroscopic improvements were investigated for single sheets of structured packing by Hu et al. (2013) and Sun et al. (2013) in absorption experiments. Advanced techniques, such as light induced fluorescence and CFD simulations, were applied by Tong et al. (2013) to investigate film flow over a triangular corrugation for different working liquids under variation of Reynolds numbers. Another field of current research is the modification of the packing surface on a microscopic scale. Kohrt et al. (2011) showed that mass transfer can be increased significantly, depending on the kind of textured surface, viscosity and Reynolds numbers. Nevertheless, these results do not allow any reliable prediction of the influence of viscosity on a column equipped with structured packing under distillation conditions.

The intention of this study is to systematically investigate the influence of viscosity and the ability of rate based models to predict the dependencies. Therefore, measurements with CB/EB as reference and a binary mixture of 2-Methyl-2-butanol and 2-Methyl-1-propanol (MB/MP) with increased viscosity were carried out. A system without polymers, or any other non-volatile components, was chosen in order to conduct experiments under total reflux, which is the standardized procedure for efficiency tests (Olujić, 2008). The utilized mixture allows an increase of viscosity by a factor of 6, in respect to CB/EB. Furthermore, the effect of the corrugation angle was investigated by conducting experiments with Sulzer Mellapak 500.Y and 500.X, the latter having a higher inclination.

The influence of viscosity and geometry on separation efficiency, quantified by the HETP value, on pressure drop and the liquid hold-up is discussed. Furthermore, results are compared with two rate-based models. The Delft model (Olujić and Behrens, 2007), (Olujić et al., 2004) is applied since it contains no empirical parameters and can represent the geometrical conditions of the used miniplant set-up. In addition, the widely used model by Rocha, Bravo and Fair (Rocha et al., 1996) is applied.

2. Experimental

2.1. *Applied test mixtures*

The influence of viscosity was determined by applying the mixture CB/EB as reference and MB/MP as viscous test mixture. The chemicals, 2-Methyl-2-butanol (purity 99+ %, extra pure) and 2-Methyl-1-propanol (purity 99%, ReagentPlus), were purchased from Acros Organics and used without further purification. Chlorobenzene (purity 99+ %) was purchased from Acros Organics and Ethylbenzene (purity ≥ 98%) was purchased from Fluka and used without further purification. Analysis via gas chromatography showed no impurities.

Viscosity is furthermore varied by changing the operating pressure. Since liquid viscosity depends on the corresponding

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