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Experimental Investigation and Design of Rotating Packed Beds for Distillation

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Improved separation efficiency and flexibility are of major importance in the downstream processing of liquid mixtures in the chemical industry, in order to respond to fluctuating demand and raw material quality. The application of the high gravity (HiGee) concept in Rotating Packed Beds (RPBs) has the potential to meet these requirements for the separation of liquid mixtures. The current work provides an analysis of the potential improvements gained by RPBs. The flexibility of RPBs is analyzed based on an experimental investigation, which evaluates the effect of rotational speed, different rotor configurations and F-factors on separation efficiency. Batch distillation experiments were performed in a single-stage pilot plant RPB with the ethanol-water system under total reflux conditions at atmospheric pressure. An analysis of the number of transfer units (NTU) reveals that separation efficiency in RPBs is not necessarily proportional to the rotational speed. There is rather an optimal rotational speed for each investigated rotor configuration. A maximum of 3.7 transfer units was achieved for a rotor with outer packing diameter of 0.36 m and an axial height of 0.01 m. Special emphasize in this experimental study is placed on the analysis of the contributions to mass transfer from the packing and the casing, which can contribute significantly to the overall mass transfer in RPBs.

1. Introduction

Distillation is one of the most important separation techniques in the chemical process industry. In conventional distillation columns, liquid flow is directed downwards due to the gravitational force, while counter-currently a vapour flows upwards. A promising way to intensify the contact between the vapour and the liquid phase is through the application of centrifugal forces in a rotating packed bed (RPB). The concept allows modifying the separation efficiency through a manipulation of the applied rotational speed. In RPBs high shear forces are provided by rotation of a rotor, equipped with a packed bed, in which gas and liquid streams are contacted in a counter-current operation. The rotor consists of an annular shaped packing surrounded by the casing as shown in Figure 1. In the RPB, the liquid is introduced at the inner periphery of the annular packing and is accelerated radially outward through an applied centrifugal field into the stationary casing. The vapour stream is introduced into the casing and flows radially inward counter-currently to the liquid due to an imposed pressure gradient. The application of centrifugal forces, 100 – 1000 times the gravitational force, generates thin films and fine droplets, thus leading to intensified contact between the phases. The potential benefits include an intense mixing, reduced equipment size, shorter residence time, use of high specific surface area packings and an enlarged operating window (Neumann et al., 2017) compared to the conventional gravity-based separation.

After the first patent on RPB by Ramshaw and Mallinson (1981), a few hundred studies have been published on various separation and reaction processes, while also several industrial applications of RPBs are recognized for example, processing of heat sensitive materials at normal atmospheric pressure due to short residence time in RPBs (Wang et al., 2011) and deaeration of liquids due to high capacity and low equipment weight of RPBs (Zheng et al., 1997). In the last three decades, various designs of RPBs were proposed and investigated, together with different mass transfer and performance models. Most of these studies attribute all of the measured mass transfer in an RPB to the packing (Kelleher and Fair, 1996), while not differentiating between mass transfer in the casing and the packing. However, in order to derive generally applicable design

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correlations for RPBs, a systematic investigation has to firstly investigate the mass transfer in different sections of RPB, before a reliable model can be proposed. Therefore, the objective of the present study is to investigate and quantify the mass transfer in these sections and study the influence of different operating parameters (e.g. rotational speed, F-factor) and rotor configurations (e.g. empty rotor, packed rotor) on the overall separation efficiency and in the specific sections. In order to highlight the importance of this differentiation, the results of this study are finally compared with reported data for RPBs and a conventional column.

2. Experiments

2.1 Experimental Setup

For the conducted experiments a specially constructed pilot-scale RPB was used. The simplified flow diagram of the experimental setup for distillation in the single-stage RPB at total reflux is shown in Figure 1. The inner diameter of the casing is 860 mm and a rotor outer diameter of 400 mm with an axial height of 10 mm was used for the experiments. The rotor had an inner diameter of 146 mm. Details of the RPB and rotor specifications can be found in the article of Neumann et al. (2016). For the experimental investigation, a metal knit mesh with a wire diameter of 0.23 mm was used as packing material. The porosity and surface area of the packing was calculated to be approximately 87% and 2500 m^2 m^{-3}. Experiments were performed with two types of rotor configurations, i.e., 400 mm OD, 10 mm axial height, with metal knit mesh packing and without packing (empty rotor). Moreover, two different F-factors, 0.35 $Pa^{0.5}$ and 0.65 $Pa^{0.5}$ at the eye of the rotor, were investigated with these rotor configurations. The F-factor is defined at the eye of the rotor as

$$F_{eye} = \frac{\dot{m}_{v,out}}{\rho_{v,out} A_{p,i}} \sqrt{\rho_{v,out}} \ , \tag{1}$$

where $A_{p,i}$ represents the cylindrical surface area at the inner radius of the packing, $\rho_{v,out}$ and $\dot{m}_{v,out}$ are the vapour density and mass flow rate at the vapour outlet of the RPB.

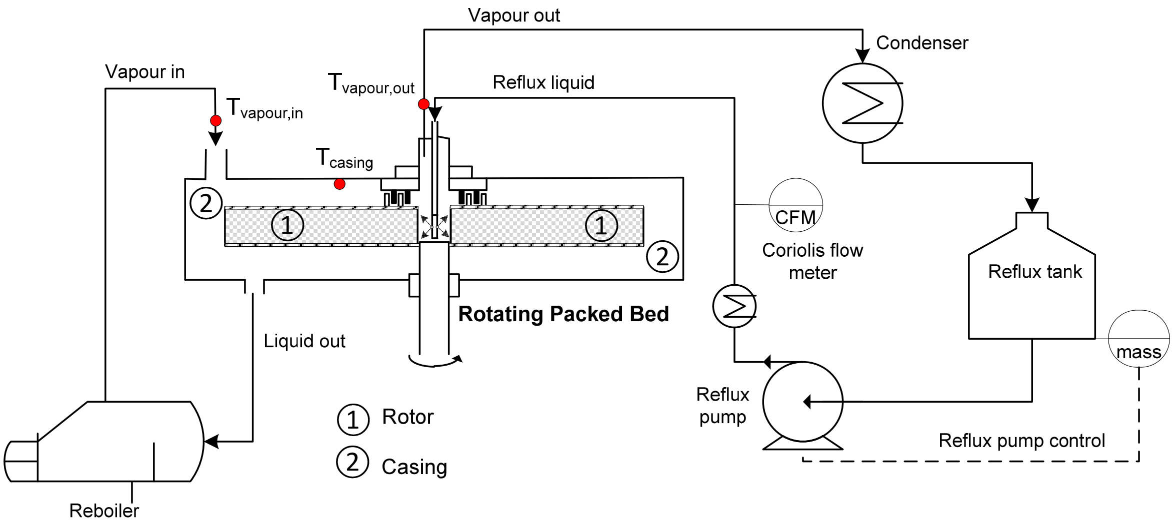

Figure 1: Simplified flow scheme of RPB pilot plant under total reflux configuration ($T_{vapour,in}$, $T_{vapour,out}$, $T_{vapour,casing}$: temperature of the vapour phase at the inlet, outlet and in the casing of RPB respectively)

2.2 Experimental Procedure

All rotor configurations were investigated by the same experimental procedure. At first, a pre-defined volume of approximately 6 liters and 10 wt.% ethanol-water was charged into the reboiler via the reflux tank. The feed was pumped from the reflux tank to the RPB, already adjusted at a specific rotational speed while exiting the RPB directly into the reboiler. The electrically heated reboiler was set to a power resulting in a F_{eye} of 0.35 $Pa^{0.5}$. Nitrogen was also fed to the reboiler as an inert gas to ensure safe operation. The vapour entered the RPB through the vapour inlet, flowing radially through the packing and leaving the RPB through the eye of the rotor. The vapour was further condensed in the overhead condenser and collected in the reflux tank. A constant mass of the liquid, collected in the reflux tank, was used to control the reflux pump to ensure operation under total reflux. A Coriolis mass flow meter was used to obtain an online measurement of the flowrate and density of the liquid reflux. Once steady state was achieved, liquid samples were withdrawn from the liquid reflux and the bottoms stream from the RPB. Steady state was assumed once the density change of the liquid reflux was less than 0.5 kg m^{-3} over 10 minutes. Samples were analysed using Mettler Toledo-Densito 30 PX. It took two hours to get the first steady state and 20 minutes to achieve new steady-state operation after modifying the rotational speed. This indicates that the RPB quickly adjusts to changes during operation. Each experimental measurement was taken twice. At first, the rotational speed was increased stepwise from 1 s^{-1} - 20 s^{-1}, while afterward the rotational speed was reduced stepwise in the same intervals.

3. Results and Discussion

Similar to the well-known HTU-NTU concept for conventional packed columns, Singh et al., (1992) introduced the ATU-NTU concept, to account for the change of fluid loading along the radius of the annular packing in an RPB. The product of the area of a transfer unit (ATU) and the number of transfer units (NTU) is determined by

$$ATU^G.NTU^G = \pi(r_0^2 - r_i^2)\,, \tag{2}$$

with r_o and r_i being the inner and outer radius of the packing. ATU^G is defined by

$$ATU^G = \frac{G}{\rho_G h K_G a}\,, \tag{3}$$

where G is the vapour flow rate, ρ_G is the vapour density, h is the axial height of the packing and $K_G a$ is the overall volumetric mass transfer coefficient based on the assumption that vapour side mass transfer resistance is dominant. In this study, ATU^G values are calculated only for comparison with reported RPB units. It is intentionally not meant for an analysis of the separation efficiency of the investigated RPB because it attributes the total separation efficiency only to the packing. Yet, it will be shown in this investigation that the casing also contributes a significant amount to the overall separation efficiency.

Based on the results of the experimental study, NTU (number of transfer units) values were calculated to quantify the effect of rotational speed (n_{rot}), F-factor (F_{eye}) and rotor configurations (packed and empty) on the mass transfer in RPBs. NTU values were calculated according to

$$NTU^G = \int_{y_{in}}^{y_{out}} \frac{1}{y - y^*} dy\,, \tag{4}$$

where y is the composition of the bulk vapour phase and y* is the composition of the vapour phase in equilibrium with the liquid phase. The integral is calculated with the limits being the vapour composition in equilibrium with the liquid in the condenser (y_{out}) and in the reboiler (y_{in}). Under total reflux conditions, the experimentally measured liquid composition is used to calculate these vapour compositions. At the operating pressure of the condenser, 101.325 kPa, the temperature, and vapour-liquid equilibrium data are computed using NRTL property method in Aspen Plus® for the ethanol-water system. Figure 2 shows the dependence of NTU on the rotational speed for the rotor packed with knit mesh (KM) and the empty rotor. Investigations on the empty rotor were performed in order to get a reference separation efficiency value. It is evident that the rotor with packing does provide better separation compared to the empty rotor.

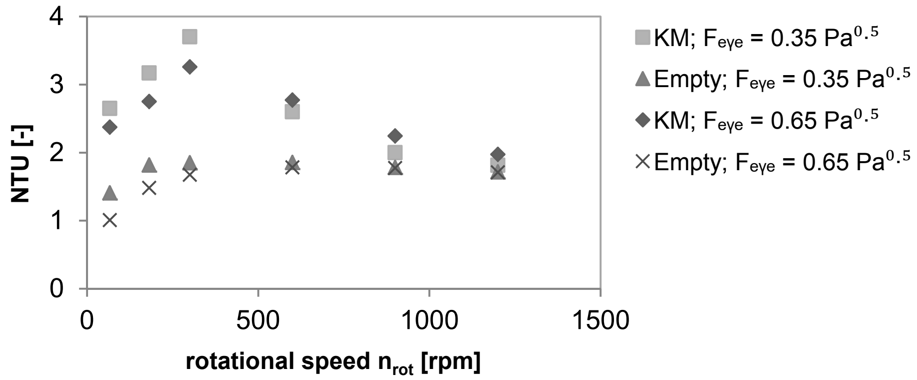

Figure 2: Effect of rotational speed on NTU at varying F_{eye} and rotor configuration (KM: rotor with knit mesh packing, Empty: rotor without packing)

The course of the NTU curves for both rotor configurations can be divided into two sections. To differentiate between both of them the optimal rotational speed (n_{opt}) is defined giving the maximum separation efficiency at the investigated conditions. In the first section ($n_{rot} \leq n_{opt}$) an increase of the rotational speed improves the separation, while in the second section ($n_{rot} > n_{opt}$) an opposite trend is observed. This suggests that for each rotor configuration n_{opt} represents the optimal rotational speed for which the maximum separation or the highest NTU value is achieved. Chu et al. (2013) also observed similar behavior with the methanol-water system in a continuous distillation two-stage countercurrent RPB (TSCC-RPB). They found the optimal rotational speed to be around 700 - 800 rpm. They associated the behavior to the interaction between contact time and effective interfacial area (Chu et al., 2013). Generation of tiny droplets at high rotational speed results in an increased interfacial area that enhances the separation efficiency, however at the same time the contact time of the phases in the packing decreases. The optimal rotational speed shows the point after which the loss of contact time overcomes the benefit of the increased interfacial area. Kelleher and Fair (1996) also observed a maximum in the NTU value for varying F-factor at a rotational speed of 500 rpm and recognized it as flooding. By analyzing an NTU-rotational speed curve at 165.5 kPa pressure, they found that there exists a maximum NTU value especially for low F-factors (Kelleher and Fair, 1996). In the current study, the maximum NTU occurs at a lower rotational speed (300 rpm) compared to the reported studies. This can be related to the applied low vapour and liquid loads compared to the size of the rotor. The investigated RPB has a higher ratio of radial length to axial height compared to the reported studies, therefore, the chances of liquid maldistribution and dry spots in the packing are higher, which leads to the loss of separation efficiency at a lower n_{opt}.
The loss of separation efficiency after a certain rotational speed is also related to the liquid dynamic hold-up in the packing which is defined as the amount of liquid inside the packing during operation (Burns et al., 2000). Burns et al., 2000, used resistance measurements to model the liquid hold-up inside the packing. They related the liquid hold-up inside the packing to different operating variables such as vapour and liquid flow rate, acceleration, and viscosity of the liquid. The model was fitted to the experimental results with the following best fit correlation (Burns et al., 2000):

$$\varepsilon_L = 0.039\left(\frac{g}{g_0}\right)^{-0.5}\left(\frac{U}{U_0}\right)^{-0.6}\left(\frac{\nu}{\nu_0}\right)^{-0.5}. \quad (5)$$

With this equation, the dependence of the liquid hold-up on liquid flow rate, acceleration and viscosity is given, whereas the influence of gas flow is assumed to be negligible. The parameters g_0, u_0, and v_0 are provided as characteristic values for RPBs by Burns et al., 2000. Although the correlation does not take into account packing properties, it is used for a qualitative check on the liquid hold-up inside the packing. The results are illustrated in Figure 3.

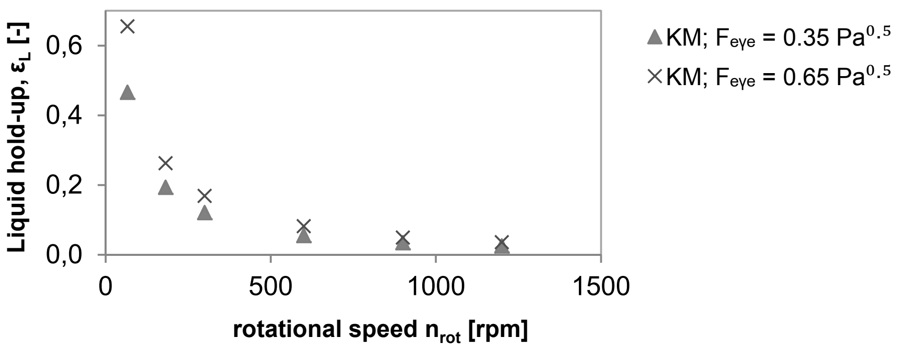

Figure 3: Effect of rotational speed on liquid hold-up at varying F_{eye}

It can be clearly seen from Figure 3 that after 300 rpm the liquid hold-up in the packing is almost negligible, resulting in a loss of separation efficiency in the packing. As expected, higher F-factors provide higher liquid hold-up inside the packing. However, the difference is almost negligible at high rpm.

Mass Transfer in the Casing

The liquid droplets leave the rotor at high speed into the casing that is completely filled with the vapour phase. Therefore, the two phases are also in contact with the casing, which contributes to the investigated total separation efficiency achieved in an RPB. Similarly, the reflux liquid sprayed on the packing at the inner periphery contacts the vapour phase leaving the rotor at the inner periphery, i.e. eye of the rotor. Most of the reported studies do not take this into account and assign the total mass transfer achieved in an RPB solely to the packing. In order to investigate the contribution of the casing to the overall mass transfer in the current RPB, the temperature inside the casing is also measured together with the vapour inlet and outlet temperatures as shown in Figure 1 (filled circles).

The overall mass transfer in an RPB can be divided into three sections, i.e. the casing, the packing and the eye of the rotor. In the current experimental investigation, the mass transfer in the packing and in the eye was lumped together due to measurement limitations of the current experimental setup. Thus, the total mass transfer in the RPB was divided into two main sections, i.e. the casing and the packing, including the eye of the rotor. The extent of mass transfer achieved in the casing and in the packing was determined according to Eq(6) and Eq(7):

$$\frac{y_{in} - y_{casing}}{y_{in} - y_{out}} = \Delta y_{casing} \quad , \tag{6}$$

$$\frac{y_{casing} - y_{out}}{y_{in} - y_{out}} = \Delta y_{packing} \quad . \tag{7}$$

In these equations y is the composition of the vapour determined from the temperature measurements at the vapour inlet, outlet and in the casing, assuming that the vapour is saturated at all time. The calculated Δy_{casing} and $\Delta y_{packing}$ are the fraction of the total mass transfer in each section respectively, determined based on the ethanol mole fraction. Figure 4 illustrates a remarkable trend of how the mass transfer is shifted from the packing to the casing at high rotational speed where almost 80% of the total mass transfer takes place in the casing. Figure 3 supports these results and the decrease of mass transfer in the packing can be attributed to the decrease of liquid hold-up in the packing with the increasing rotational speed. Furthermore, higher F-factors benefit the mass transfer in the packing at high rotational speed as compared to the mass transfer in the casing due to the generation of the higher interfacial area in the packing.

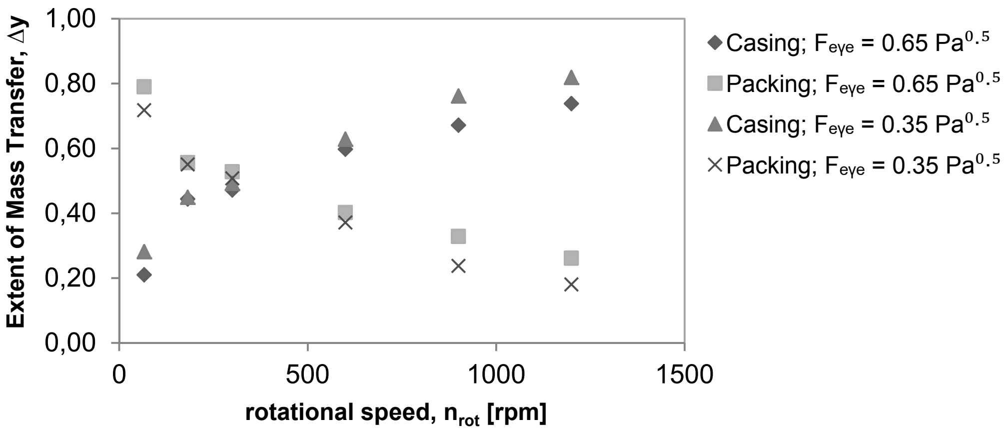

Figure 4: Effect of n_{rot} on fraction of mass transfer in the casing and in the packing at varying F_{eye}

These experimental results lead to the interesting conclusion that for an efficient operation of an RPB, the design of the packing plays an important role along with the axial height, outer and inner diameter in achieving the desired separation efficiency. The separation efficiency of the packing can be improved by improving the wettability of the packing, resulting in the higher interfacial area, and also by improving the design of the packing to provide higher contact time between the phases. Figure 4 gives an insight into the packing revealing that operation at higher F-factors leads to an improvement in the efficiency of the packing compared to the casing. Moreover, the existing correlations for the overall mass transfer in RPBs need to be further developed to incorporate the mass transfer in the casing.

Comparison with Other RPBs and Conventional Column

To allow for a comparison with reported experimental investigations for RPBs in the literature, the ATU-NTU concept is applied. ATU values are calculated according to Eq(2) and NTU is defined by Eq(4). Table 1 shows that the calculated NTU and ATU values of the current study are comparable to those reported in different literature references. Thus the obtained results are likely transferable to the RPBs investigated in these studies. For a comparison with a conventional distillation column, the HETP-concept is utilized.

Table 1: Comparison of calculated NTU and ATU values with the reported literature data

Reference	NTU [-]	ATU [m^2]
Current study	1.8 – 3.7	0.03 – 0.05
Kelleher and Fair (1996)*	1.5 – 6.5	0.04 – 0.13
Nascimento et al. (2009)*	0.69 – 1.69	0.01 – 0.027

*Values are estimated from the published data

With this concept, the height equivalent of a theoretical plate (HETP) is calculated dependent on the number of theoretical stages, N_{th}, and the height of the packing. In the case of RPBs, the height of the packing is taken equal to the radial length of the packing (Mondal et al., 2012) and the HETP value of the investigated packing is found to be approx. 0.045 – 0.09 m. For a conventional column with RMSR25-3 packing (a_p = 191 m^2 m^{-3}), HETP values for the considered ethanol-water system were found to be approx. 0.25 – 0.28 m (Markos, 2011). Hence, a 3 – 5 fold size reduction is achieved with the investigated operating conditions in the pilot scale single-stage RPB. The improvement in terms of the HETP value of the RPB can be attributed to the significantly higher specific surface area of the knit mesh packing. It is important to point out that a more realistic comparison of the conventional column with the RPB will result by taking volume of the packing and the mass transfer in the casing into account while computing HETP values for RPBs.

4. Conclusions

In this study, a systematic design procedure for distillation operation in RPBs was initiated by experimentally investigating the distribution of mass transfer among different sections of RPB. It is found that for the investigated F-factors, an optimal rotational speed exists (300 rpm for the current set-up) giving the maximum separation efficiency and after this optimal rotational speed, loss of separation efficiency results due to loss of liquid hold-up and contact time in the packing. Furthermore, experimental investigations revealed that the casing contributes a significant fraction, more than 60 % at speed higher than the optimal rotational speed, in the overall mass transfer. Therefore, the existing mass transfer correlations need to be decomposed accordingly, in order to account for the contribution of the casing. Future work is needed to investigate the overall and sectional separation efficiency for different packing designs and higher F-factors to exploit the available large operating window of the pilot scale RPB and to build an experimental database to develop design guidelines.

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