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Abstract

Heat pumps can be used to efficiently reduce the energy requirements in distillation columns by 50-80%. In conventional methods like Vapor Compression (VC) and Vapor Recompression (VRC) the required temperature lift generated by the compressor is determined by the temperature difference between reboiler and condenser. This limits the number of applications as economic temperature lifts are typically restricted to 30 °C. A way to circumvent this limitation is not to recompress the vapor from the top, as in a VRC, but extract the vapor at a lower stage, recompress this vapor and transfer the heat via internal heat exchangers from the rectifier to the stripper stages.

Heat integration in distillation columns (HIDiC) by means of a compact plate-fin heat exchanger (PFHE) further reduces the energy consumption because of the low pressure drop and the small minimum approach temperature, which can be in the order of 1 K due to the high specific surface area of 1,000-1,500 m²/m³. In a previous study (Hugill et al, 2007) it was shown how the PF-HIDiC concept can be converted into an industrial application leading to substantial savings in utility and investment cost.

For the model system cyclohexane/n-heptane the performance of a 1 meter long PF-HIDiC has been quantified with respect to hydrodynamics, separation and heat transfer. The experimental number of equilibrium stages of 1.6 is lower than 2-2.5 as predicted by the Delft model. However, the main objective, achieving separation in a PF heat exchanger was fulfilled. The heat transfer coefficient was found to be equal for the stripping and the rectifying mode; a value of 145±11 W/m²/K was found, which is 25% lower than the design value. No influence of the operation mode on the separation efficiency was observed. A pressure drop of up to 5 mbar/m was found, which is higher than predicted. Flooding was not observed at any of the process conditions.

Special attention was given to the design and manufacturing of the liquid distributor because of the strong effect of maldistribution on the separation efficiency. In a separate set-up the distributor was tested for the model system n-decane/N₂. Within its design limits of an F-factor between 1.0 and 2.8 Pa^{0.5} the maldistribution fraction, f_{av} =0.12±0.03.

Introduction

Distillation is the main separation technology in refineries and the chemical process industry, because of the attractive purification characteristics, the high

production capacity and turndown ratio, and the straightforward design procedures. More sophisticated techniques have become state of the art to handle streams with less favorable thermodynamic properties, in particular small relative volatilities and azeotropic mixtures. The high energy demand in bulk distillation columns (1-100 MW) and the low thermodynamic efficiency of less than 10% (Humphrey, 1991) remain the major drawbacks. A number of improvements have been developed over the years directed at reducing both operating and capital cost.

In extractive distillation (ED) a solvent or separating agent is added in order to increase the relative volatility of the components to be separated. In azeotropic extractive distillation the separating agent is used to break the azeotrope. As a consequence the reflux ratio, column diameter and reboiler duty can be reduced and/or the column height can be lower. Commercial low volatility solvents include sulfolane, triethylene glycol (TEG), NMP and NFM (Lee, 2000). The recovery cost of the solvent is an integral part of the economy of extractive distillation processes. Kossack et al (2008) have developed a systematic method for molecular modeling based entrainer selection and optimization of the ED flow sheet. ED is particularly effective for relative volatilities below 1.2. Industrial examples of ED processes are purification of aromatics in petrochemistry, butadiene recovery in naphtha cracking and separation of cycloparaffins from naphtha (Doherty and Knapp, 2004).

Instead of affecting the thermodynamics of the system also selection of the column internals is a way to increase distillation efficiency. Random and structured packings with specific surface areas from 250 up to 900 m²/m³ are continuously being improved with the objective to optimize stage height, pressure drop, liquid load, and turn down ratio. The main recent advancements in tray columns focus on high-capacity trays with centrifugal devices or structured packing demisters although at the cost of an increased pressure drop (Olujic et al, 2009).

Since the 1980's dividing wall columns (DWC's) have been introduced which allow the separation of three component feeds in a single column leading to interesting reductions in both energy consumption and investment cost. Recently even more complex DWC's have been constructed to separate four component mixtures in pure products (Olujic et al, 2009).

In contrast to improvements of the VLE or the column internals, both inside the column, a number of energy reducing measures can be considered outside the column by addressing the reboiler and condenser. These include side reboilers (Bandyopadhyay, 2007) dephlegmators (Bakke, 1997, and Wang and Smith, 2005) and heat pumps (Null, 1976). Side reboilers use waste heat at a lower temperature than the bottom reboiler and thus increase the exergetic efficiency. Dephlegmators or reflux condensers are compact heat exchangers, such as PFHE's, used to reduce energy consumption in low temperature gas separations. The final objective is to apply heat integration in order to reduce utility cost effectively to zero (Linnhoff et al, 1983).

Heat pumps lift the temperature level of the vapor in order to use this as the heat source for the reboiler. Vapor recompression columns, VRC's, with a compressor as heat pump, are the 1st generation heat pump distillation systems shown in **Figure 1**. VRC's have been introduced since the 1980's for close boilers with high reflux ratio's and subsequently high reboiler/condenser duties.

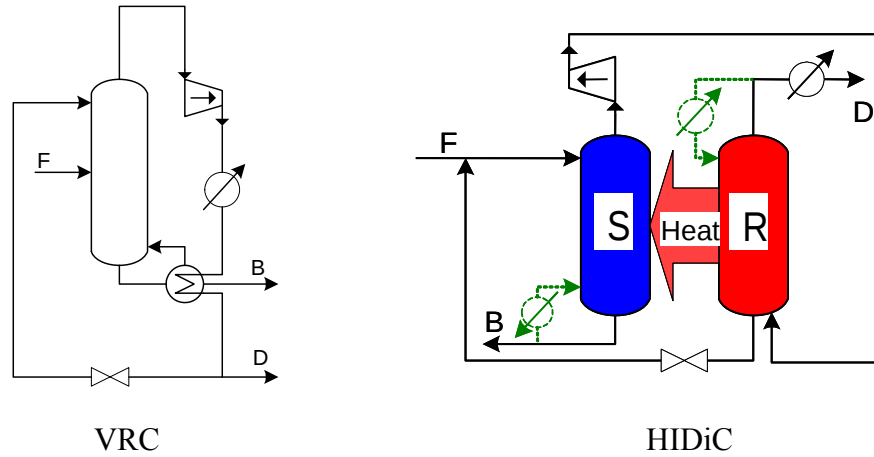


Figure 1. The Vapor Recompression Column (VRC) and the Heat Integrated Distillation Column (HIDiC)

In order to improve the energy savings and the applicability of heat pumps in distillation the Heat Integrated Distillation column (HIDiC) concept, also shown in Figure 1, was introduced by Mah et al (1977) and further developed in Japan (Aso, 1998, Nakaiwa, 2003) and in the Netherlands at TUDelft and ECN (Olujic, 2003, Hugill, 2005). Process analysis shows that this 2nd generation heat pump distillation system can have additional energy savings in the order of 30-50%, depending on the separation target (Olujic, 2009).

Different HIDiC configurations have been patented, including the concentric tray, CT-HIDiC (De Graauw et al, 2003) and the plate-fin, PF-HIDiC (Hugill, 2003). This paper is the first publication of experimental results for the PF-HIDiC.

In the PF-HIDiC a plate-fin heat exchanger (PFHE) is modified to obtain channels with counter-current flow of gas and liquid, resulting in separation. As in conventional PFHEs, the hot and cold streams are separated by plates. In a HIDiC, the hot stream is the rectifier section and the cold stream is the stripper section (Gadalla, 2005). The concept is shown in **Figure 2**.

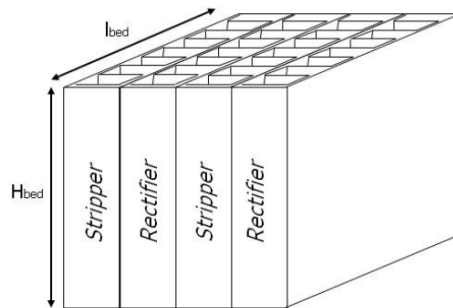


Figure 2. Schematic representation of the channels in a PF-HIDiC.

In this paper the design aspects will be discussed for the distributor, the PFHE and a proof-of-principle test unit with a nominal capacity of 50 kg/h. Experimental data on distribution, pressure drop, separation performance, heat transfer, and turndown ratio will be compared with the design models.

Experimental set-up

In order to assess the operating window and performance of the PF-HIDiC principle a test unit was constructed with a single process layer for a nominal capacity of 50 kg/h for a binary cyclohexane/n-heptane feed, to be operated under total reflux.

The unit consists of a plate-fin heat exchanger, a distributor and auxiliary equipment. Details of the unit design will be discussed first.

The PF-HIDiC test unit

The PF-HIDiC test unit, shown in **Figure 3**, contains the following sections:

- A reboiler circuit with a sump vessel, V-103, a recirculation pump, P101, and heat exchanger, E-101, with controlled steam supply. A safety release valve (not shown) can be opened to remove the liquid to a safety vessel, V-101.
- A 3-layer plate-fin heat exchanger (PFHE) with distributor discussed in more detail below.
- A heating/cooling oil system with circulation pump connected to the two outer layers of the PFHE, E-103.
- A condensation circuit consisting of a condenser, E-102, a condensate vessel, V-102, and a reflux pump, P-102. Temperature sensor TIC-106 is used to control the cooling water flow.
- A N₂ pressure control system to purge the set-up and maintain a slight overpressure of 1.1 bar in order to prevent air entering the system.
- Liquid sampling systems in the reboiler, SP-102 and the condensate circuit, SP-101, for off-line GC analysis.
- The two main sensors for process analysis are the reflux flow sensor, MFI-102, the pressure drop sensor, dPI-101 and the temperature of the heating bath oil, *TI-402 – TI-407*.
- A data acquisition system that continuously monitors and stores flows, pressures and temperatures.

The unit can operated in adiabatic and diabatic modes. The former means using the external section as insulation only, while diabatic operation allows running the test section as a rectifier (external cooling) or a stripper (external heating).

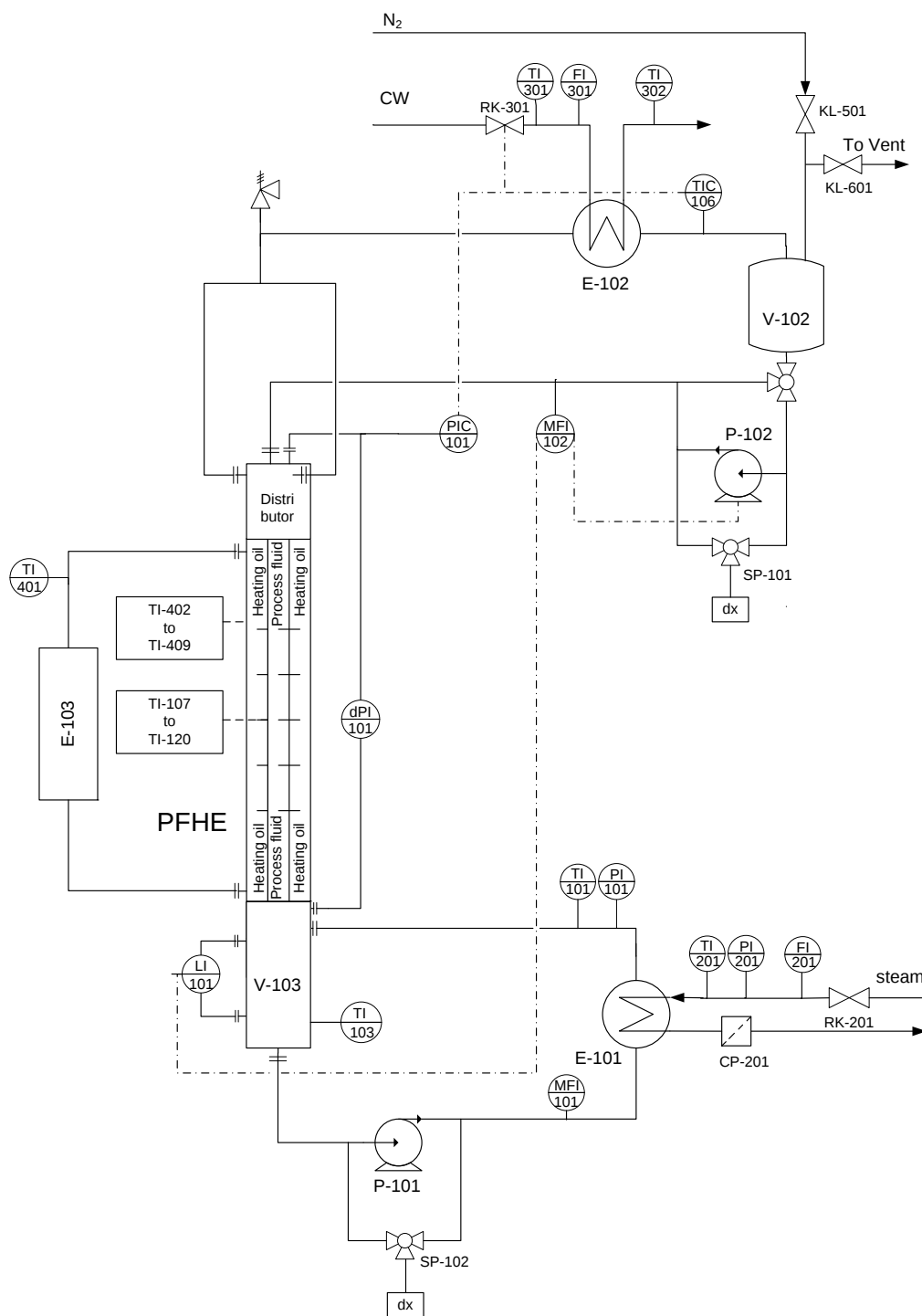


Figure 3. Flow diagram of the PF-HIDiC test unit

In this study the composition was kept constant at a cyclohexane mole fraction of 0.72. Typical operating conditions for the PF-HIDiC are given in **Table 1**.

Table 1. Experimental ranges for the PF-HIDiC

Feed composition	0.72	mol/mol	Reboiler duty (max)	6	kW
Operating pressure	1.1	bar	PFHE duty ($\Delta T = \pm 10$ K)	± 2	kW

In **Table 2** the relevant physical properties of the two compounds have been summarized.

Table 2. Physical properties of the test system

Physical property (at b.p., 1.1 bar)	cyclohexane	n-heptane
Vaporization enthalpy (kJ/kg)	350	325
Liquid density (kg/m ³)	715	625
Vapor density (kg/m ³)	3.2	3.9
Relative volatility (average)	1.6	

The plate-fin heat exchanger (PFHE)

The PFHE consists of a 3-layer sandwich. The distillation process (countercurrent vapor/liquid flow) occurs in the central/process layer, which is connected to the reboiler and the condenser. The two outer layers of the sandwich are for flow of cooling or heating fluid. The test unit does not simulate a complete HIDiC, but rather one half of a HIDiC, either the rectifier (with external cooling) or the stripper (with external heating).

The fins in the process layer are perforated plain fins while the outer layer fins are off-set fins. The main dimensions of the distillation core layer are given in **Table 3**. These dimensions result from the analysis of the performance of the module as predicted by the simulation model developed by Hugill and van Dorst (2005).

Table 3. Dimensions of the process layer. Nominal capacity 50 kg/hour.

Plate height	1	m	Plate thickness	1	mm
Plate width	0.2	m	Plate gap = fin height	15	mm
Cross sectional area	0.0028	m ²	Fin spacing = fin width	4.8	mm
Heat transfer area*	1.60	m ²	Number of fin channels	40	-
Specific area, S	528	m ² /m ³			

*: this is the metal area “seen” by the vapor; effectively the area is about 0.4 m²

PFHE's with specific surface areas of $S=1,500 \text{ m}^2/\text{m}^3$ for boiling hydrocarbons typically have heat transfer coefficients of $530 \text{ W/m}^2/\text{K}$ (Shah and Sekulić, 2003). However, for the PF-HIDiC heat transfer is not the only criterion. Therefore the design is based on the optimization of heat transfer, separation, wetting, hydraulic diameter and pressure drop for a feed of 50 kg/h, resulting in a PFHE with a relatively low specific surface area $S=528 \text{ m}^2/\text{m}^3$. The fin height of 15 mm is relatively large; in commercial PFHE's the fin height usually is in the range 4-8 mm. The design model predicts for this PFHE a heat transfer coefficient $U=200 \text{ W/m}^2/\text{K}$.

The selection of the off-set fins in the outer layer is connected to the requirement that the heat transfer in the outer layer is not limiting. It was calculated that higher heat transfer coefficients can be obtained with off-set fins compared to plain fins. The geometry of the offset fins was optimized to maximize heat transfer.

The fins in the process layer protrude ca. 10 mm at the lower end of the core (this allows better detachment of the liquid flow, and increases the capacity in the condensation case) and also at the upper end (this allows up-flowing vapor to avoid the liquid distributor).

The distributor

The PFHE contains a large number of parallel vertical channels separated by fins and plates. This implies that redistribution, even with the perforated fins, hardly occurs. Several authors have shown that maldistribution in packed columns can seriously decrease the separation performance. Billingham and Locket (2002) used the parallel column model to determine the maximum value for the maldistribution fraction, f_{max} , beyond which the HETP increases rapidly. For a column with N equilibrium stages:

$$f_{max} = \frac{y_{N+1} - y_N}{y_N - y_o} + \frac{x_1 - x_o}{x_{N+1} - x_o} - \left(\frac{y_{N+1} - y_N}{y_N - y_o} \right) \cdot \left(\frac{x_1 - x_o}{x_{N+1} - x_o} \right) \quad (1)$$

In this equation the compositions y_N , x_{N+1} , y_o and x_1 are specified in the separation target and the remaining y_{N+1} and x_o follow from the equilibrium equations:

$$y_{N+1} = \frac{\alpha x_{N+1}}{1 + x_{N+1}(\alpha - 1)}; \quad x_o = \frac{y_o}{y_o + \alpha(1 - y_o)} \quad (2)$$

For the cyclohexane/n-heptane model system used in this study both f_{max} and the stage efficiency, E , are given in Figure 4 for the separation target ($x_D=0.95$; $x_B=0.05$) for N=4.

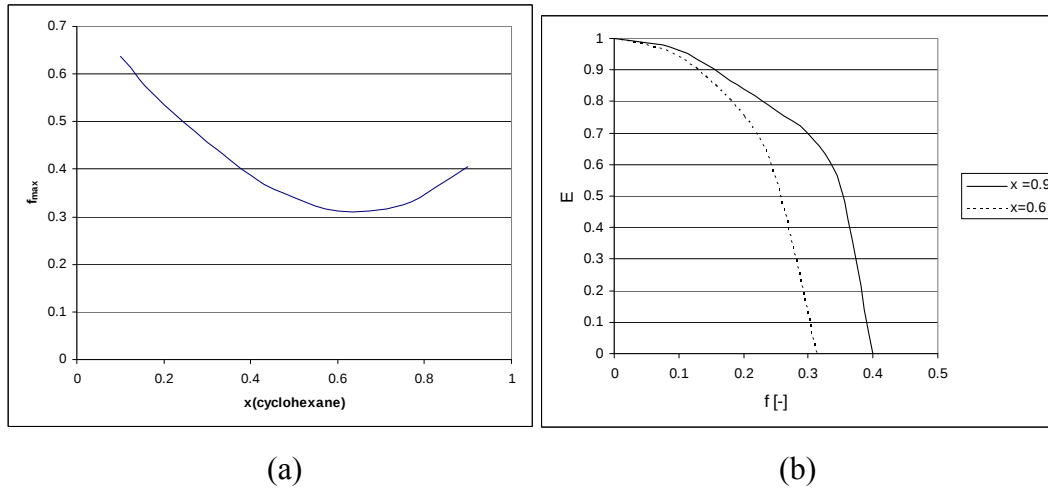


Figure 4. Maximum maldistribution fraction as a function of feed composition (a), and stage efficiency as a function of maldistribution fraction for $x_F=0.6$ and 0.9 (b)
Conditions: cyclohexane/n-heptane, $x_D=0.95$; $x_B=0.05$, $N=4$, $p=1.1$ bar

Figure 4a shows that the maldistribution fraction should be kept below 0.3, in particular for $x_F=0.6$. From Figure 4b it can be concluded that if the stage efficiency should be above 90%, f_{max} should rather be smaller than 0.13.

With this in mind the distributor was designed using the design rules of Bonilla (1998). Because of the sensitivity of the PF-HiDiC configuration and the flexible turndown ratio the trough distributor with calibrated orifices was selected for a cyclohexane/n-heptane feed of 50 kg/h.

The main distributor dimensions are presented in **Table 4**. Details of all distributor dimensions are given by Saric et al (2010).

Table 4. Dimensions of the distributor. Nominal capacity 50 kg/hour.

Number of orifices	14	-	Maximum liquid height above orifices	180	mm
Orifice diameter	1.5	mm	Theoretical turndown ratio	3	-

Results and discussion

Distribution

A number of distributor configurations were qualitatively tested using visual inspection. The final distributor has a central liquid holder with two arrays of orifices. The liquid jets are further distributed by means of 2 splash plates. Before entering the PFHE the liquid passes a fine metal mesh. This distributor was tested quantitatively and optimized in the distributor test unit (DTU) using n-decane and N₂ as testing system. The distribution results are expressed as the average maldistribution fraction, f_{av} , and were determined for different F-factors under simulated total reflux conditions. This means that with increasing n-decane feed rate the N₂ feed rate is also increased such that the molar flow rates of N₂ and n-decane are always equal. The results are presented in **Figure 5**.

The F factor is the common parameter to determine the hydrodynamic operating range for distillation columns and is defined as:

$$F = u_o \sqrt{\rho_{vap}} (Pa^{0.5}) \quad (3)$$

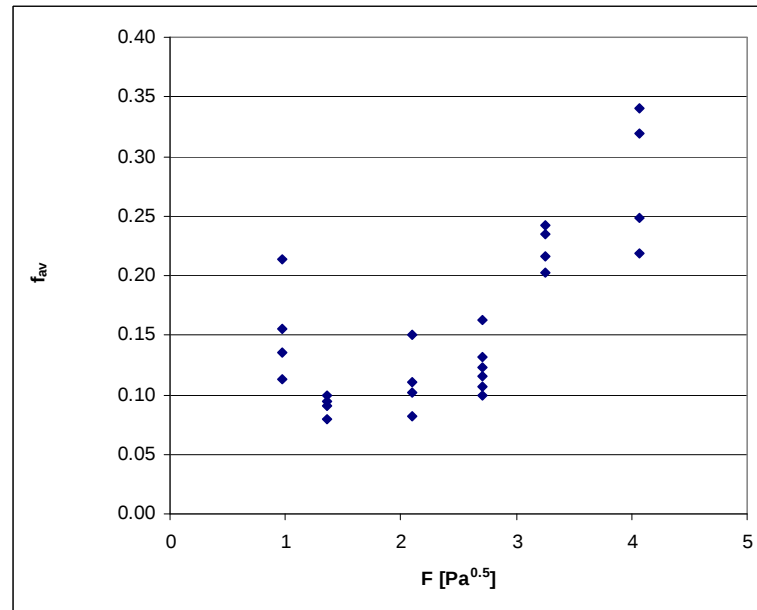


Figure 5. Average maldistribution fraction at different F factors. n-decane/N₂ under simulated total reflux conditions

The f_{av} -curve shows a minimum value of $f_{av}=0.12\pm0.03$ in the range $1.0<F<2.8$. Note that the design capacity of 50 kg/h corresponds with $F=2.7 \text{ Pa}^{0.5}$. In other words the distributor turndown ratio equals 2.8. f_{av} increases below $F=1.0$ due to the fact that the jets do not properly hit the splash plates. The increase in f_{av} for $F>2.8$ is due to flooding in the PFHE section below the distributor, which is also visually observed.

Operation, control and heat transfer

In the PF-HIDiC the vapor flow rate and thus the F-factor at the bottom of the PFHE is independently controlled by adjusting the steam control valve to the reboiler. With the equipment operated under total reflux and in the adiabatic mode, the vapor flow rate is the same as the reflux flow rate measured with MFI-102. The results, for $x_B=0.72$ mol/mol, in **Figure 6** show that the boil-up, expressed as F-factor, increases linearly with the percentage of steam valve RK-201 opening. The design target for the PFHE and distributor of 50 kg/h corresponding with an $F=2.7 \text{ Pa}^{0.5}$ is obtained at 62% steam valve opening. The lower distributor limit where jet breaking will occur is at $F\text{-factor}=1.0 \text{ Pa}^{0.5}$ or 42% steam valve opening.

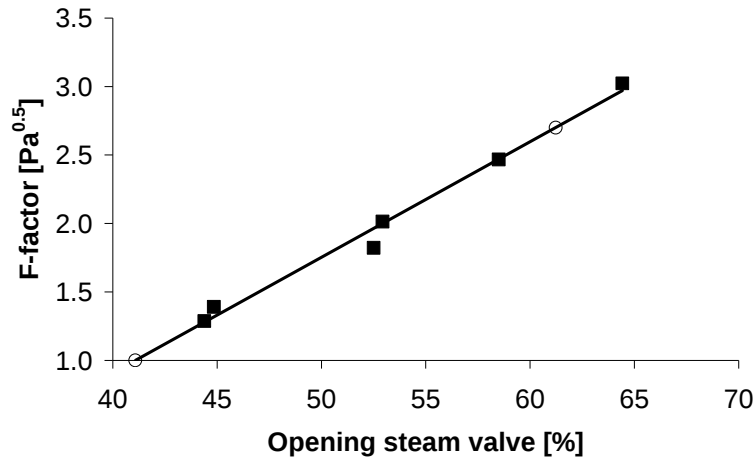


Figure 6. Control of the reboiler at adiabatic and total reflux conditions.
 $x_B=0.72$ mol/mol, $\circ$: distributor design F-factors

The adiabatic operation mode, as in **Figure 6**, is achieved by preventing any flow of the thermostat oil. Due to the insulation of the set-up the temperature of the mantle becomes constant and equal to the process layer.

Figure 7 shows the results for the diabatic total reflux conditions, where the oil temperature is either above or below the process layer temperature, thus simulating the stripping or the rectifying mode, respectively. The selected temperature difference ranges are typical for industrial applications of the PF-HIDiC.

In diabatic operation the F-factor changes over the length of the PFHE, therefore the value at the top, F_{top} , is given. The F-factor at the bottom of the PFHE is equal to the value for diabatic operation at $\Delta T=0 \text{ K}$.

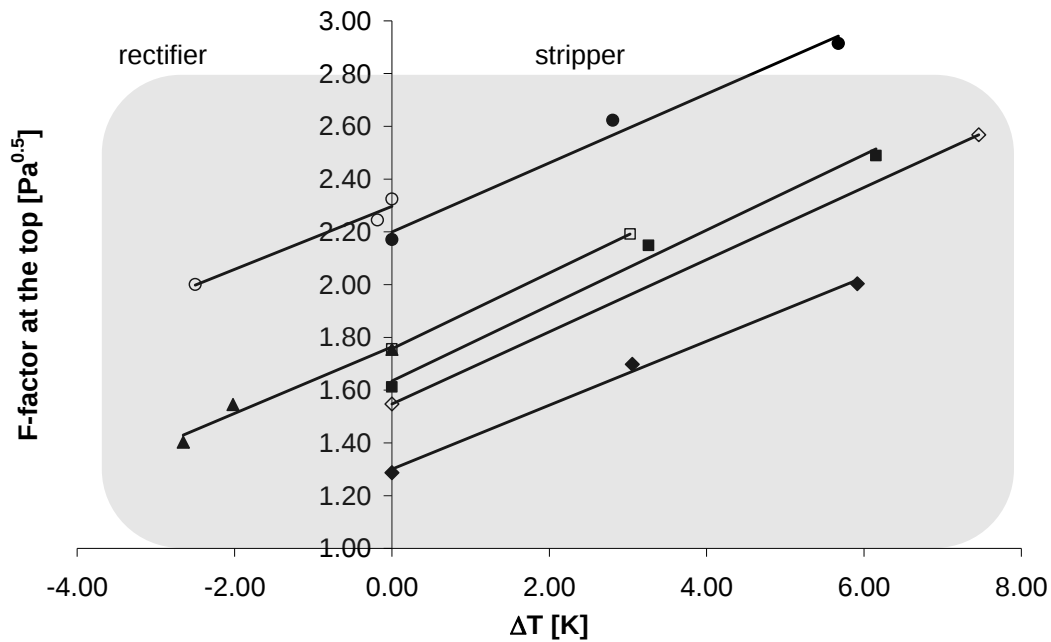


Figure 7. Measured F-factor at the top of the column for different bottom F-factors and temperature differences with the thermostat oil

As the PFHE should be operated at $F > 1.0$ to avoid jet breaking in the distributor and at $F < 2.8$ to avoid flooding, the operating range for the PF-HiDiC is as indicated by the shaded area.

The fact that the lines are linear and parallel indicates that the heat transfer coefficient in the PFHE remains more or less constant for different F-values and both operating modes. The heat transfer coefficients for different F-factors at the bottom of the column are given in **Figure 8**.

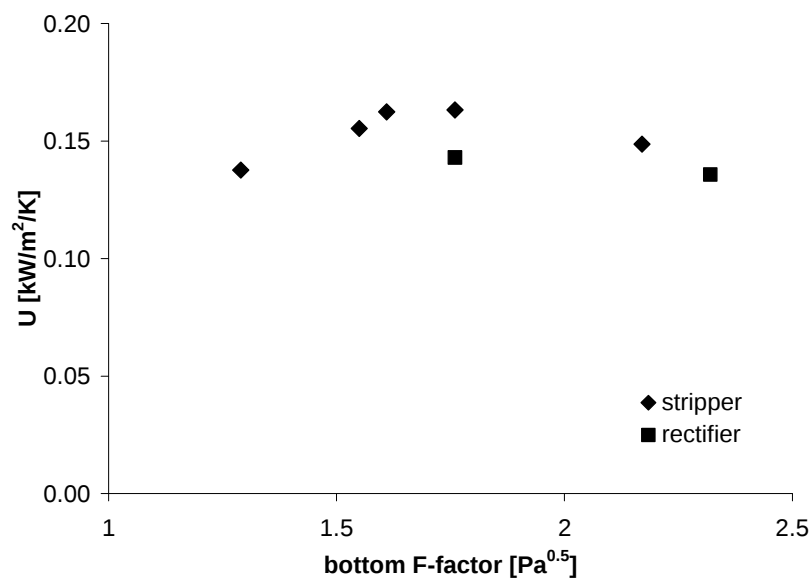


Figure 8. Measured overall heat transfer coefficient of the PFHE for stripping (heating) and rectifying (cooling) mode.

A heat transfer coefficient $U=145\pm11$ W/m²/K is calculated, based on the 1.60 m² heat transfer area. This experimental value is about 25% below the design value.

It is interesting to note that the difference of the overall heat transfer coefficient between rectifier and stripper operation is within the error margin. This might indicate a good distributor performance. In case of poor liquid distribution a higher heat transfer coefficient might be expected for rectifier operation due to improved wetting.

Separation

One of the main challenges of this project is achieving separation in a PF heat exchanger. The extent of separation was measured by GC analysis of the liquid streams of the reflux and the bottom stream. At an average relative volatility, $\alpha=1.6$, slightly depending on the composition, the number of equilibrium stages was calculated with the Fenske equation:

$$N = \frac{\log \left[\frac{x_d}{1-x_d} \cdot \frac{1-x_b}{x_b} \right]}{\log [\sqrt{\alpha_{top} \cdot \alpha_{bottom}}]} \quad (4)$$

The number of equilibrium stages for both diabatic and adiabatic experiments is shown in **Figure 9**, together with the curve based on the Delft model (Olujić, 2004). The bottom F-factor is used in Figure 9 since these values correlate slightly better than those for the top F-factor. For modeling adiabatic conditions were assumed.

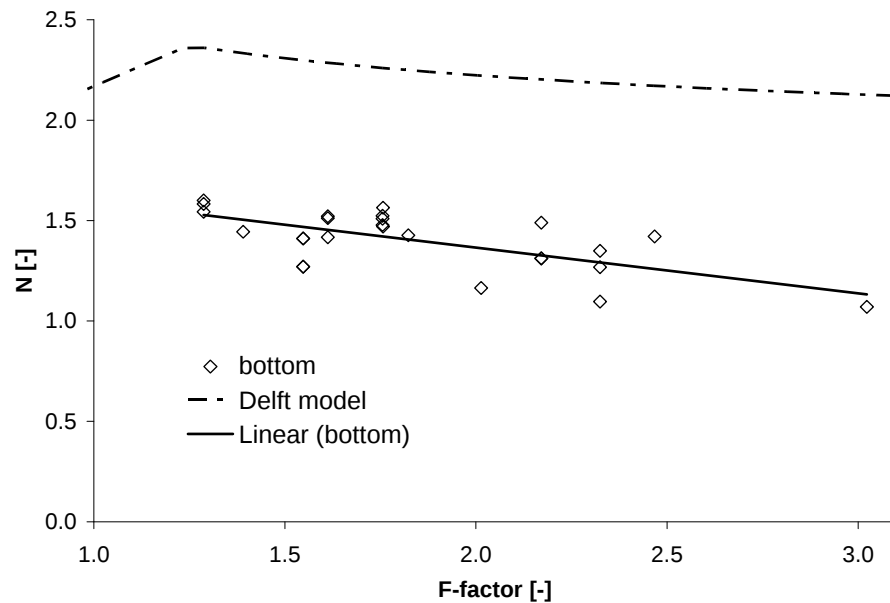


Figure 9. Number of equilibrium stages at different bottom F-factors for both experiments and model (Olujić, 2004)

The number of equilibrium stages of less than 1.6 is low compared to the 2-2.5 as predicted by the Delft model. Certainly, if one takes into account that the reboiler section also contributes to the separation. However, the trend of decreasing separation

at higher F-factors is similar in both direction and slope and separation in a modified PF heat exchanger has been successfully demonstrated.

The relatively low separation efficiency is possibly caused by maldistribution of the liquid. Therefore the extent and the effects of maldistribution will have to be investigated further for the PF-HiDiC.

There is no clear drop in the separation efficiency at higher F-factors indicating that no flooding occurs at even the highest F-factors.

No clear effect of the operating mode (adiabatic, stripping or rectifying) on the separation efficiency was observed.

Pressure drop

The pressure drop measured for increasing F-factor, both under adiabatic and diabatic conditions is presented in **Figure 10**, together with three modeled pressure drop curves.

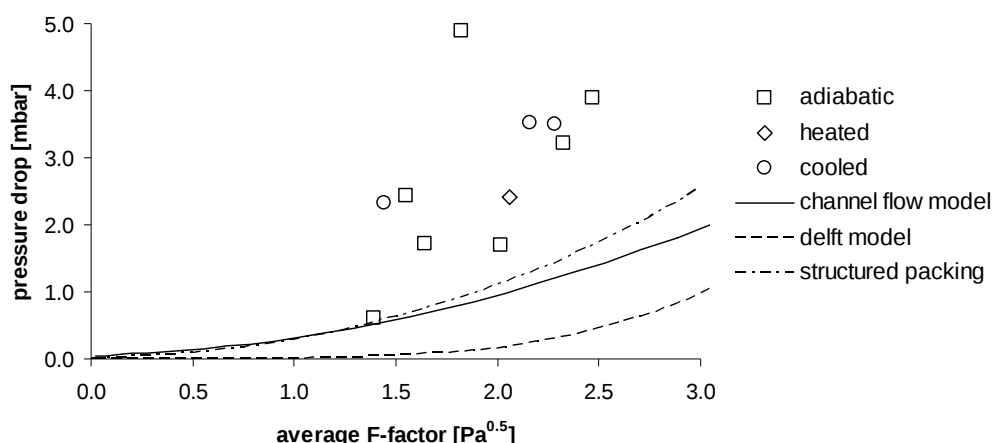


Figure 10. Pressure drop at different F-factors for adiabatic and diabatic operation.

The measured pressure drops are higher than predicted by the channel flow model (Hesselgreaves, 2001) and the Delft model for both the 90° PFHE and a 60° structured packing with equal dimensions (Olujić, 2004).

The scatter in the data can be attributed to the relatively small pressure drop over the 1 meter set-up. The experimental pressure drop also incorporates pressure drop in the distributor while this is not taken into account in the models. However, the pressure drop over the distributor is expected to be on the low side.

Even at the highest F-factors no flooding was observed, indicating a high possible vapor load for a PF-HiDiC.

Conclusions

A plate fin heat exchanger was successfully modified into a PF-HiDiC and the performance of the set-up was studied with a model system cyclohexane/n-heptane.

The main objective, achieving separation in a PF heat exchanger was successful. However, the number of equilibrium stages less than 1.6, was lower than 2-2.5 as predicted by the Delft model. No influence on the separation efficiency was observed when changing from the rectifying to the stripping mode.

The pressure drop was found to be up to 5 mbar/m, which is higher than predicted. However, flooding was not observed even at the highest F-factor of 2.5.

The overall heat transfer coefficient was found to be 145 ± 11 W/m²/K, 25% lower than the design value. The heat transfer coefficients are the same, within the experimental error, for the stripping and the rectifying modes.

The effect of maldistribution on the separation efficiency can be severe and should be a focus of attention in the design of a PF-HiDiC. The liquid distributor for the set-up was designed for an F-factor between 1.0 and 2.8. Within these limits, the maldistribution fraction, $f_{av} = 0.12 \pm 0.03$.

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