CHEMICAL ENGINEERING TRANSACTIONS

VOL. 69, 2018

A publication of AIDIC
The Italian Association of Chemical Engineering
Online at www.aidic.it/cet

Guest Editors: Elisabetta Brunazzi, Eva Sorensen

ISBN 978-88-95608-66-2; **ISSN** 2283-9216

Heat Integrated Distillation Column (Hidic): Experimental Study on a New Concentric Column Technology

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Distillation is the most applied separation technology. Its major drawback is the low thermodynamic efficiency (typically around 15 %). In response to the environmental issues that concerns energy consumption of distillation column, HIDiC (heat integrated distillation column) which combines advantages of vapor recompression and diabatic operation, is expected to have a large impact on energy saving. In this study, a concentric column which contains an innovative column packing designed in LGC research lab of Toulouse is carried out. First of all, this novel technology is characterized from a heat transfer point of view in a dedicated pilot plant. Compared to the Raschig super-ring results, the heat transfer in this innovative column packing is much more efficient, with a heat transfer increase of 102 %. A second HIDiC pilot has been implemented to test the operability and the overall performances of this HIDIC technology. Different tests have been made to reach a time of steady state of 90min. Several experiments were carried out with different operating parameters (boiler heat and pressure ratio). The Cyclohexane/n-Heptane binary mixture is used for these experiments. The simulation results of a classical distillation column are compared to HIDiC column steady state operation. This comparison demonstrates an energy saving about 40 %.

Distillation column, Heat integration, Heat Integrated Distillation Column, Energy efficiency, Process control, Operability.

1. Introduction

Distillation is a unit operation, which is widely used in separation processes. It represents approximately 40% of the total energy of the chemical process industry and more than 50% of the operating cost of the plant. Generally, capital and operating costs increase when the relative volatility decreases, then, the energy consumption increases (Kiss et al, 2014). In literature, a promising solution aims to reduce the energy consumption is proposed and is called Heat Integrated Distillation Column (HIDiC) (Figure 1-b) (Rijke et al, 2007).

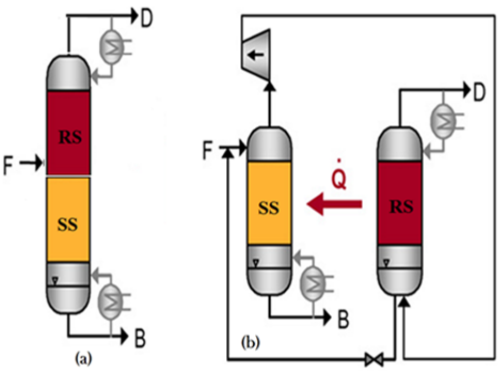


Figure 1: a. Conventional distillation column, b. HIDiC.

The rectifying section and the stripping section are separated. Heat is transferred inside the distillation column from the rectifying to the stripping section by a heat transfer technology, because the operating pressure (and thus the temperature) of the rectifying section is increased by means of the compressor. In this paper, there are two objectives, to characterize and validate the heat transfer in an innovative column packing in order to use it as a technology of heat transfer between two columns and to prove the operability of the HIDiC technology with a pre-industrial pilot plant.

2. Material and methods

2.1 Pilot of heat transfer validation

An experimental pilot is carried out in our laboratory to validate a new technology of heat transfer. Indeed, the heat transfer of aluminium foam packing (Figure 2-b) is compared to Rashig Super-Ring column packing (Figure 2-a). The heat transfer is done through the wall enhanced by fin effect due to this new technology. The foam packing is manufactured by a foundry process and is designed specifically for our HIDiC application (porosity control, structural regularity, and other confidential structural parameters) and developed in Laboratory of Chemical Engineering (LGC of Toulouse).

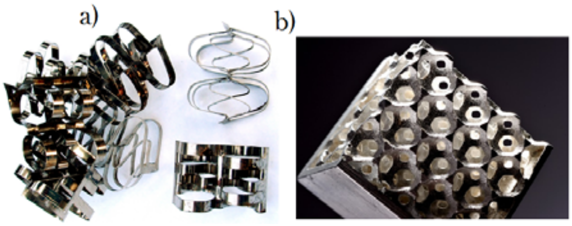
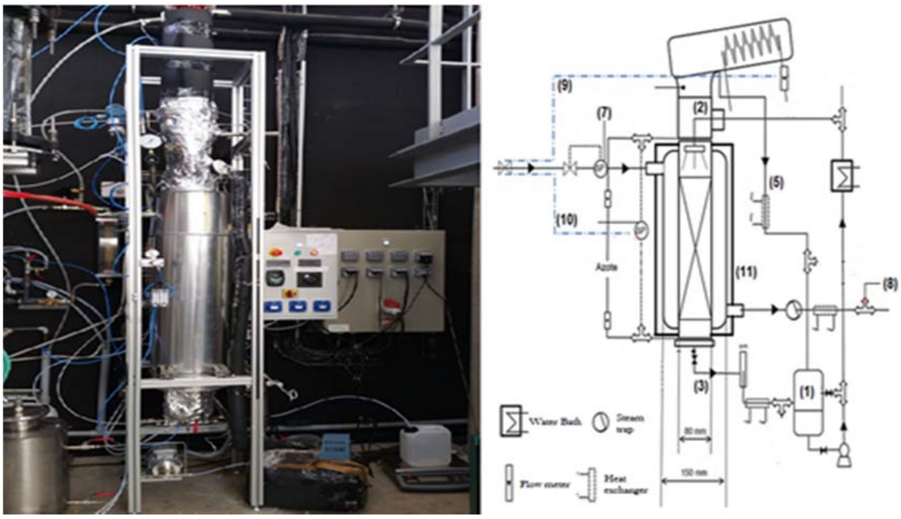


Figure 2: a) Raschig Super-Ring, b) Metal foam packing.

An experimental setup (Figure 3) is designed as a co-current heat exchanger with a concentric column, which contains packing 1 m height. The extern diameter and the intern diameter are 150 mm, 80 mm respectively. Two fluids flow in co-current, Steam in the extern column and pure butan-1-ol in the intern column. A condenser, where coolant water flows, is installed in the top of the concentric column in order to condensate the vapour of pure butan-1-ol.
The heat exchanged is calculated too by the flow rate of coolant water, input temperature and output temperature of coolant water. The thermal conductance can also be calculated by the heat exchanged and the temperature difference between the steam in the extern column and pure butan-1-ol at boiling point in the intern column.

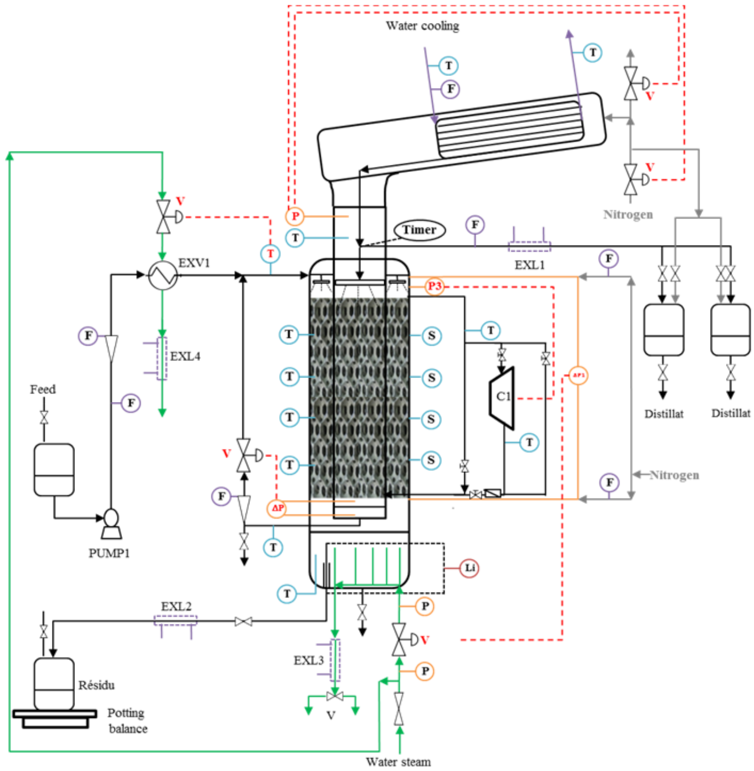


(1) Feed
(2) Distributor
(3) Residue
(5) Distillate
(7) Gauge
(8)Steam condensate
(11) Insulation

Figure 3: Experimental set-up of heat transfer validation.

2.2 HIDiC pilot of operability validation

A second pilot is design with a pre industrial size. It is designed with a concentric column, which contains 2 m height packing. The extern diameter and the intern diameter are 150 mm, 80 mm respectively. Aluminum foam packing is installed in the inner and extern column. These are the same as those designed and used previously in the first pilot presented. The Figure 4 presents the scheme of the pre-industrial pilot plant with the different utilities flow.



T: temperature Measures
P: pressure Measures
S: sampling
ΔP: pressure drop measures
F: flow measures
PUMP1: feed pump
C1: compressor
EXV: vapour heat exchanger
EXL: liquid heat exchanger
V: valve
Timer: reflux timer for inner column

Control loop
Water steam flow
Nitrogen flow
Vapor flow
Process flow

Figure 4: pre-industrial HIDiC pilot plant scheme

The regulation of the pilot is insured by different control loop. The Table 1 present the principles of this control loops the action variables, the controlled variables and some remarks.

Table 1: List and principles of HIDIC pilot control loops

Action variables	Controlled variables	Remarks
Steam flow in the feed preheat exchanger	Feed temperature	Control the feed temperature
Steam flow in the reboiler	Pressure drop in the extern column	Control the steam flow in the extern column
Feed and discharge of nitrogen in the inner column	Pressure in the inner column	The opening and closing of two valves are controlled by the pressure measure in the inner column.
Compressor frequency	Pressure in the extern column	The objective is to compress the vapors leaving the extern column without inducing a vacuum.
Liquid flowrate of the bottom inner column	Liquid level in the bottom of the inner column	Controlled the liquid level in the bottom inner column

3. Results and discussion

When the temperatures are time-independent and the steady state is obtained, the measurements are able to be operated. The thermal and mass balances are verified. The reliability of experimental results is proved by a repeatability test. The ΔT represent the temperature difference between the intern and extern column.

3.1 Comparison of two packing technologies in the pilot of heat transfer

On the Figure 5, the heat transferred determined by the pilot containing Raschig Super-Ring is compared to the heat exchanged obtained with Metal foam packing. The heat transferred of both technologies is linearly proportional with mass flow rate of the intern column liquid. The figure above shows that the transferred heat values of metal foam packing are bigger than the Raschig Super-Ring values. It means that the innovative column packing is more efficient than the Raschig Super-ring in heat transfer. Thus, the average gain obtained in heat transfer of the metal foam packing is 102%. The heat transfer in the column containing Raschig super-ring is largely compromised due to thermal resistance occurrence. Contrary to Raschig super-ring, the metal foam packing is moulded directly on to the inner wall.

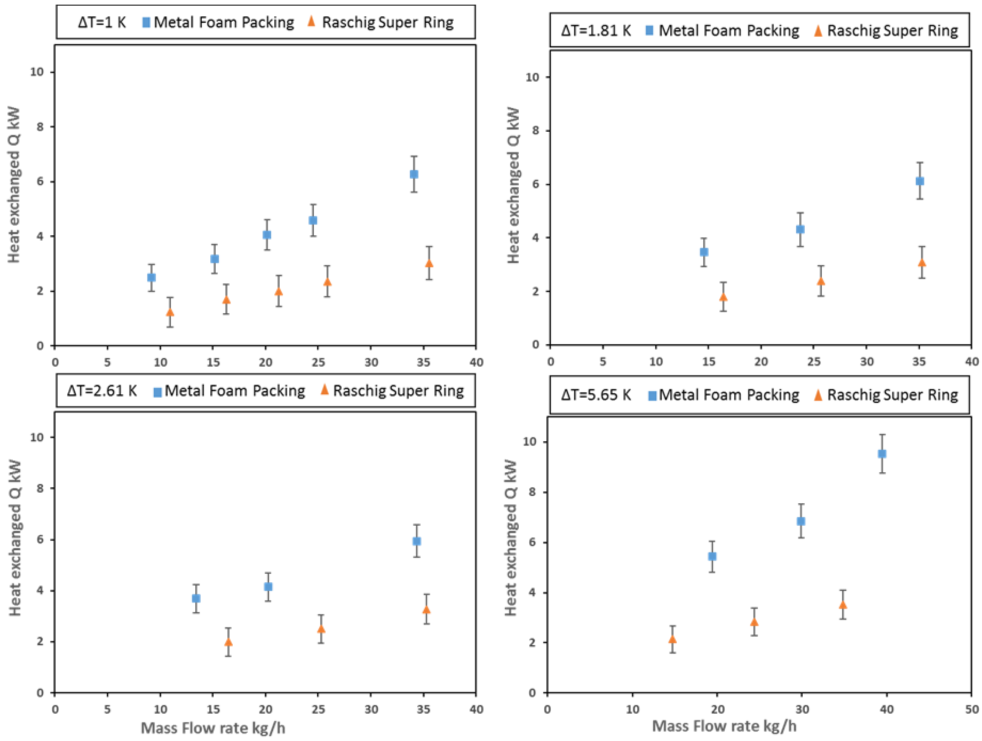


Figure 5: Comparison of the two technologies of heat transfer.

3.2 Thermal conductance of Metal foam packing

The obtained thermal conductance UA (W / K) is plotted as a function of the temperature difference for different intern liquid flow rates (Figure 6).

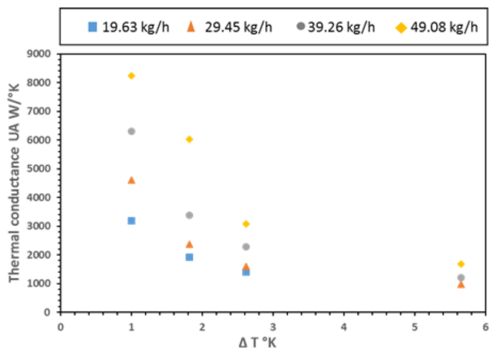


Figure 6: Variation of thermal conductance of the pilot containing metal foam packing with temperature difference.

The first observation is the thermal conductance decrease with the temperature difference between inner and extern column increase. This can be explained by the increase of film liquid thickness in the extern column and thus the thermal resistance, when the steam condensation is more significant due to the temperature difference augmentation. The second remark is that the thermal conductance values are the most significant with the biggest intern liquid mass flow rate (49.08 kg/h). It means that the increase of the feed flow allows having a better wetting, regular and as homogeneous on the metal foam packing surface, thus, the increase of the liquid-packing contact improves the heat transfer. In contrast, a low watering rate leads to the appearance of dry zones. Therefore, the obtained thermal conductance of the metal foam packing is 1285 W/K. Contributions to heat transfer resistance from outside, inside and column wall are respectively 67%, 29% and 4%.

3.3 Operability of the HIDiC technology, experiments on the pre-industrial pilot plant

With this pilot plant, several experiments are performed to prove the operability of the HIDiC technology. Different operating parameters can be set up depending on the desired separation. The Table 2 summarizes the operating parameters and the feed distilled with the HIDiC.

Table 2: Operating parameters of HIDiC plant

Feed	Inner column pressure	Reflux**	Compressor	Reboiler
11kg/h *$X_{cyclohexane}$ = 50% *$X_{n\text{-}heptane}$ = 50% Temperature: 15°C	Pint = 1.5 bar	No reflux, total discharge to the distillate	Control the extern column pressure fixed at Pext= =1.01bar.	Heat at the reboiler is around 0.57kW

**: mass fraction **: The pressure difference and thus temperature difference between the inner column and the outer column causes the liquid descending in the outer column is vaporized, and the upward vapor in the inner column is condensed. This intern reflux is significant and sufficient for the desired separation so the reflux is zero.*

This experiment is completed to prove the operability of the HIDiC technology. Several experiments are performed to find the good start procedure and minimize the setting of the pilot. With this procedure, a steady state is reached, around 90min. This experiment demonstrates that the HIDiC technology is industrially operable with a good industrial piloting and control/command. The unmeasured variables are calculated with an experimental validation model. Each stream is characterized by its mass flow in kg / h, its mass composition and its temperature. These parameters with the different powers and the pressures of each column constitute the variables of the system. In order to make the measurements more reliable and to determine the unmeasured variables, we have implemented a data validation procedure which consists in correcting the raw measurements of the sensors in order to satisfy equations of a model while remaining close to the raw measurements. The problem thus formulated can be written in the form of a problem of optimization of a quadratic criterion with nonlinear constraints. The Table 3 presents the mass balance of the chosen HIDiC experiment.

Table 3: experiment HIDiC mass balance

	F	D	W
Flowrate (kg/h)	11.03	5.36	5.63
*$X_{cyclohexane}$ (-)	0.49	0.79	0.22
*$X_{n\text{-}heptane}$ (-)	0.51	0.21	0.78
T (°C)	15	97.23	92.45
global mass balance		0.28%	

*: mass fraction

The recovery ratio of cyclohexane at the distillate and n-heptane at residue is respectively 78% and 79%. The results are compared to the simulation of a classical distillation column and a HIDiC simulation with the same separation specifications. The simulation is performed on ProsimPlus. The Table 4 present the results of these simulations, especially the energy consume by the different process and compared to the energy consumption of the experimental HIDiC.

Table 4: energy consumption comparison between the different separation processes

	HIDiC experimental	Distillation simulation	HIDiC simulation
Q_b (kW)	0.55	1.20	0.60
P compressor (kW)	0.21	-	0.12
Reflux rate (-)	-	1.27	0.01
Total energy	0.76	1.20	0.72

Qb: reboiler power

On the one hand, the comparison with classical distillation point out the energetic gain is around 40%. Actually it is difficult to add electric energy with thermal energy. The weighting factor depends on the way in which this energy is produced. Taking for France, a cost of 9cts € / kwh for electricity and 5.5cts € / kwh for thermal heat, the cost reduction is still 25%. This comparison confirmed that HIDiC technology reduces energy consumption for this case study. On the other hand, a simulation model for HIDiC is made to predict or validate experimental parameters. Only the energy consumption was presented here but all the parameters fit between experimental results and simulation results (Table 5 and 6).

Table 5: mass balance comparison between experimental and simulated HIDiC

	F		D		W	
	experimental results	simulation results	experimental results	simulation results	experimental results	simulation results
Flowrate (kg/h)	11.00	11.00	5.36	5.38	5.63	5.62
Xcyclohexane (-)	0.49	0.49	0.79	0.78	0.22	0.21
Xn-heptane (-)	0.51	0.51	0.21	0.22	0.78	0.79
T (°C)	15	15	97.23	97.12	92.45	92.90

For the mass balance comparison, the difference between experimental and simulation value don’t exceed 5% with a mean difference of 2%. Thus, the simulation is able to reproduce the behaviour of the column, allows to determine the desired operating parameters for a given separation and validate the experimental measurements.

Table 6: energy consumption comparison between experimental and simulated HIDiC

	experimental results	simulation results
Q_b (kW)	0.55	0.61
Q_c (kW)	0.51	0.54
Q_{exch} (kW)	0.829	0.828
P compressor (kW)	0.21	0.12

Q_b: reboiler power / Q_c: condenser power / Q_{exch}: power exchange between intern and extern column

For the energy consumption difference, this is due to the heat loss overestimated on the experimental pilot for the reboiler energy. The difference for the compressor energy is due to the isentropic, mechanical and electrical factor that underestimated the compressor energy for the simulation results.

4. Conclusions

In perspective to find a new technology of heat transfer and to have a high performance in heat transfer between rectifying column and stripping column, an innovative column packing is studied and compared to Raschig Super-Ring. The heat transfer in the metal foam packing is more efficient with a gain mean of 102 % on heat transfer. The obtained thermal conductance UA of metal foam packing is 1285 W / K.

Experiments on the second HIDiC pilot reveal that HIDiC technology is industrially operable with rational setting time and sufficient steady state long time. It also proves an energy saving of around 40% with an innovative column packing, compare to the simulation of a classical distillation column with same separation specification. More HIDiC experiments with classical column packing must be performed to quantify the proportion of energy saving caused by the HIDiC technologies and the innovative column packing. Moreover, a simulation model of HIDiC allow to predict, to verify or validate experimental data, measured or unmeasured.

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