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Experimental Evidence on Performance of an Advanced Gauze Structured Packing under Deep Vacuum Distillation Conditions

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Results are presented of total reflux experiments carried out at Fractionation Research Inc. with Montz-Pak A3-500M, an advanced corrugated sheet wire gauze packing, using paraxylene/orthoxylene system at 0.02 and 0.1 bara, respectively. In both cases an expectedly good performance was achieved, ranging from eight stages at low- to four stages per unit bed height at high vapor loads. Interestingly, within preloading region the packing exhibited better efficiency at 0.02 bara, however this was at the expense of somewhat increased pressure drop, and, strikingly, no gain on capacity side. Delft model proved capable of capturing observed trends, but exhibits a pronounced discrepancy with respect to measured efficiency and pressure drop at 0.02 bara, and suggests a higher flooding limit than observed.

1. Introduction

In order to facilitate fractionation of heavy molecules such as various free fatty acids and the like, the lowest industrially viable operating pressures (well below 0.1 bara at the column top) are utilized in industrial practice. These applications are generally referred to as deep vacuum distillations. Here the tolerable amount of pressure drop is rather low. Therefore the structured packings with lowest pressure drop per stage, i.e. conventional and advanced versions of well-established corrugated sheet wire gauze packings are a preferred choice.

A relatively large pressure drop as encountered in these demanding separations is a major design and operation concern, and reliable experimental data is required to validate properly the predictive models used for column design/rating purposes. However, adequate experimental data are very scarce and those obtained at large enough scale using Fractionation Research Inc. (FRI) facilities in Stillwater, Oklahoma, and that available at Bayer Technology Services (BTS) in Leverkusen, Germany, in conjunction with well-established low relative volatility test systems were limited to 0.1 bara top pressure. The opportunity to move into unexplored operating pressure range emerged upon decision of FRI membership to choose Montz-Pak A3-500M for a Category 1 test in 2015. Montz delivered packing and auxiliary equipment and FRI made all necessary equipment and manifold modifications including installation of internal bayonet type condenser, to be able to run total reflux distillation experiments at a top pressure of 0.02 bara. Upon initial operational difficulties followed by thorough analysis and subsequent dedicated refinements of design and layout of some auxiliary internals and operating procedure stable enough operation at such a low pressure was ensured and planned tests carried out.

In these first-ever tests carried out with a gauze packing at FRI, a good overall performance was achieved. However, compared to base FRI test at 0.1 bara, data obtained at 0.02 bara indicate some peculiarities, i.e. a significant efficiency gain accompanied by a pronouncedly increased pressure drop within preloading region, and, most surprisingly, no capacity gain.

In absence of any experimental reference for 0.02 bara data, the results have been evaluated and interpreted using Delft model (Fair et.al., 2000), which, as proven in previous BTS and FRI studies with advanced Montz packings (Olujić et al., 2012, 2013, 2015), captures well the observed trends and approaches closely

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measured efficiencies and pressure drop at 0.1 bara, but fails to do this properly at 0.02 bara, indicating that the onset of loading and flooding at this pressure should occur at higher vapor load than observed.

2. Experimental

The packing tested was Montz-Pak A3-500M, an advanced version of common type corrugated sheet wire gauze packing, with unperforated surface and a smooth short bend at lower end of corrugations (see photo in Figure 1a). Nominal specific geometric area of this perforated wire gauze packing is 500 m^2/m^3, however based on measured corrugation dimensions of delivered packing the installed area is 478 m^2/m^3. The corrugation inclination angle is 60^o with respect to horizontal and the void fraction (porosity) of the dry packed bed is around 0.92 m^3/m^3. Packed bed comprised 13 layers of packing. Each layer consisted of four segments and was rotated by 90^o to the previous one, and the total installed bed height was 2.2 m.

(a)  *(b)* 

Figure 1 – Photographs of a segment of Montz-Pak A3-500M (a) and the liquid distributor used in this study (b), both courtesy of Dr. T. Cai of FRI

Total reflux experiments have been conducted utilizing low pressure column with internal diameter of 1.22 m. The test system was paraxylene/orthoxylene (px/ox), and the operating pressures employed were 0.1 bara and, for the first time, 0.02 bara. As mentioned before, to enable operation at 0.02 bara the low pressure distillation column within FRI plant was adapted accordingly. A bayonet-type condenser was installed in higher, 2.44 m internal diameter section, and the condensate was collected in a basin installed below the condenser and via a downpipe delivered to the distributor. The liquid scrapped from the section walls was collected in the vane type liquid collector placed above a narrow trough liquid distributor type S, installed at the top of lower, 1.22 m internal diameter section. A photograph of the distributor is shown in Fig. 1b. This specially designed, very large turndown distributor (0.3 – 5 m^3/m^2h) had 80 uniformly distributed drip tubes, and underneath each of these was installed a cap containing multiple capillary working extensions (“drip fingers”) providing in total 1120 drip or pour points, ensuring, as proved in a dedicated water test at both Montz and FRI, a good performance over the whole range of operation at both pressures.
Pressure drop is measured directly, and is based on readings taken from pressure taps located above and below the bed. Measured packing efficiency is expressed as the height equivalent to a theoretical plate (HETP), and for each data point it is determined from the ratio of the bed height and the number of theoretical plates or equilibrium stages observed at given operating conditions. The Fenske equation was used to determine the number of equilibrium stages based on compositions, i.e. mole fractions of light component in the liquid samples taken from distributor and/or basin and from a cross-sampler placed immediately below the bed, and the corresponding relative volatility. All data were taken for each vapor load within given range twice, with an interval of approximately one half of hour. A more detailed description of the test setup and procedures as employed in a typical total reflux structured packing test at FRI can be found elsewhere, e.g. Olujić et al. (2013).

3. Results and Discussion

3.1 Experimental evidence

Figure 2a shows the efficiency (HETP) and pressure drop of A3-500M as a function of the vapor load, i.e., so called F-factor (F_G), as measured with px/ox system at 0.1 bara. As expected, the efficiency of this corrugated sheet structured packing made of wire gauze is at its best at low end, where a separating power equivalent to 8 equilibrium stages per unit bed height is developed. It tends to deteriorate gradually with an increasing vapor load, i.e. increasing specific liquid load, reaching some 5 stages around the point of onset of loading.

Regarding the high level of performance achieved in present FRI test, it reflects closely that obtained at the same pressure (dashed lines shown in Fig. 3) in a BTS test carried out at same pressure in a 0.59 internal diameter using another well-established low relative volatility test system, i.e. chlorobenzene/ethylbenzene (Olujić et al., 2012). Such a good agreement between efficiencies and pressure drops measured at FRI and BTS at 0.1 bar is not surprising because the same was observed in tests conducted earlier with Montz-Pak B1-250MN (Olujić et al., 2013) and B1-350MN (Olujić et al., 2015).

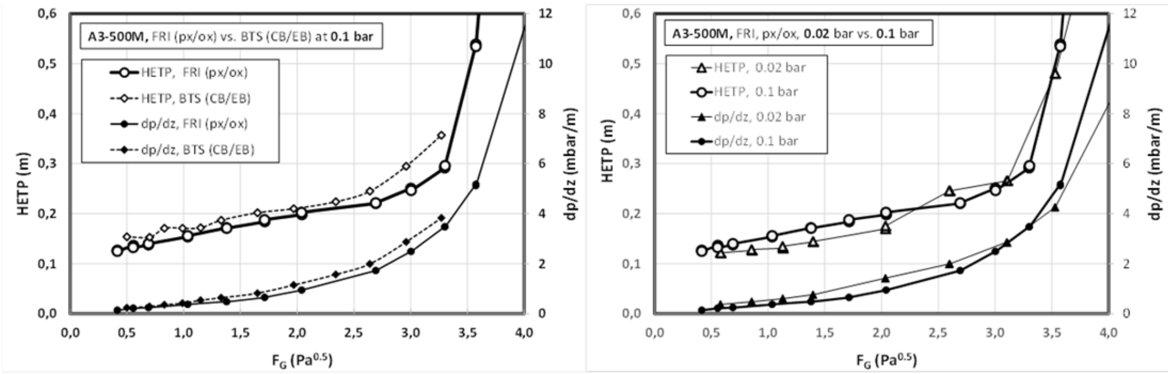

Figure 2 – Efficiency and pressure drop of A3-500M as measured at 0.1 bara with px/ox system at FRI and CB/EB system at BTS (a) and the effect of pressure on efficiency and pressure drop for px/ox system (b).

Figure 2b shows comparison of performance of A3-500M at 0.1 bar with that obtained at a five times lower operating pressure (0.02 bar) at the column top. The HETP and pressure drop curves obtained at 0.02 bara exhibit similar trend, with exception of one point within loading region, and lie close to those obtained at 0.1 bara. In both cases HETP value tends to increase gradually until a point is reached (onset of loading) upon which the HETP curves overlap and become steeper and this trend is more or less preserved until the flood point, i.e. the F-factor at which the efficiency disappears suddenly. Peculiarly, this sudden departure of efficiency (flooding) occurs in case of 0.02 bara operation at a somewhat lower vapor load and pressure drop (< 3.0 mbar/m compared to ~3.5 mbar/m at 0.1 bara). Note that FRI column is still operable beyond the point of departure in efficiency, and the hydraulic flood, i.e. complete loss of operability, occurs usually at a pressure drop close to or above 10 mbar/m.

If we take a closer look at the curves shown in Figure 2b, we see that data obtained at 0.02 bara indicate a significant efficiency gain accompanied by a pronouncedly increased pressure drop within preloading region. Within loading region the efficiency curves overlap, while those of pressure drop overlap close to flooding limit, and beyond that point that obtained at 0.1 bar exhibits higher values and a steeper increase in pressure drop. This may also be considered to be peculiar, because in previous tests carried out with sheet metal packings there was no such a pronounced pressure effect on efficiency and even less on pressure drop within preloading region.

Without proper experimental reference, we have used the Delft model (DM) in conjunction with average values of relevant physical properties of the liquid and vapor (Table 1) to facilitate evaluation and interpretation of 0.02 bara efficiency and pressure drop data.

3.2 Delft Model based considerations/evaluations

Basically, A3-500M is a corrugated sheet structured packing with given nominal area and corrugations inclined to horizontal by 60^o, including a smooth bend at lower end. With corrugation dimensions known, DM does not require any adjustable empirical parameter to arrive at the predictions of efficiency and pressure drop at given operating conditions. However, A3-500M differs from common sheet metal packings. It is made of woven wire gauze and its surface exhibits at low liquid loads a strong capillary effect facilitating a much better wetting of installed surface area than achievable with sheet metal packings. With this in mind, in the present case the empirical correction term that in DM accounts for less efficient use of area in case of sheet metal large specific geometric area packings, described in detail elsewhere, e.g. Olujić et al. (2004), is omitted. According to the adopted model, the fraction of installed area covered by a flowing liquid film depends strongly on the liquid load and, consequently, it is larger at larger operating pressure and in a total reflux experiment it tends to increase proportionally to the increase in vapor load (see right hand side of Table 1).

Table 1: Representative values of physical properties of px/ox system and the relation between specific liquid load and vapor load at two operating pressures as employed in this study.

Test system	Paraxylene/Orthoxylene		F_G ($Pa^{0.5}$)	u_{Ls} (m^3/m^2h) 0.02 bara	u_{Ls} (m^3/m^2h) 0.1 bara
Pressure (bara)	0.02	0.1	0.5	0.72	1.39
Temperature (^oC)	43	74	1	1.43	2.79
Liquid composition (-)	0.49	0.54	1.5	2.15	4.18
Liquid density (kg/m^3)	851	823	2	2.87	5.57
Liquid viscosity (Pa s)	5.39 E-4	3.91 E-4	2.5	3.59	6.97
Liquid diffusivity (m/s^2)	2.36 E-9	3.57 E-9	3	4.30	8.36
Vapour density (kg/m^3)	0.115	0.406	3.5	5.02	9.76
Vapour viscosity (Pa s)	6.63 E-6	7.30 E-6	4	5.74	11.15
Vapour diffusivity (m/s^2)	7.66 E-5	2.37 E-5			
Surface tension (N/m)	0.0268	0.0234			
Relative volatility (-)	1.287	1.240			
Equilibrium line slope (-)	0.99	0.98			

A comparison of predicted and measured efficiencies (*HETP* values) and pressure drops for 0.1 bara and 0.02 bara is shown in Figures 3a and 3b, respectively. Predicted *HETP* curves exhibit similar trend as measured ones, and within model validity range (preloading region where liquid film flow prevails) are in both cases on the safe side, while the discrepancy between predicted and measured values is more pronounced in case of 0.02 bara operation. Note that opposite to experiment DM suggests a somewhat higher packing efficiency in case of 0.1 bara operation. This is not surprising if we consider that model simply translates the effect of pressure on physical properties and governing variables via the working expression representing packing efficiency. Note that in the present case (a low relative volatility test system) the *HETP* is identical to the height of the overall gas phase transfer unit (HTU_{Go}), which, per definition, is the ratio of superficial vapor velocity (u_{Gs}) and the product of the overall vapor phase mass transfer coefficient (k_{Go}) and the effective area (a_e):

$$HETP = HTU_{Go} = \frac{u_{Gs}}{k_{Go}\, a_e} \tag{1}$$

The latter known generally as volumetric mass transfer coefficient assumes in present case a factor 1.7 higher value at 0.02 bara, because the increase in vapor diffusivity and consequently in the value of vapor mass transfer coefficient is much stronger than the decrease in specific liquid load when the operating pressure drops from 0.1 to 0.02 bara. However, with a nearly factor two larger superficial velocity of ascending vapor at 0.02 bara, the resulting efficiency is somewhat lower, i.e. corresponding *HETP* curve lies above that representing 0.1 bara operation. Therefore the discrepancy between predicted and measured *HETP* curve at 0.02 bara, the latter lying below that of 0.1 bara, is more pronounced.

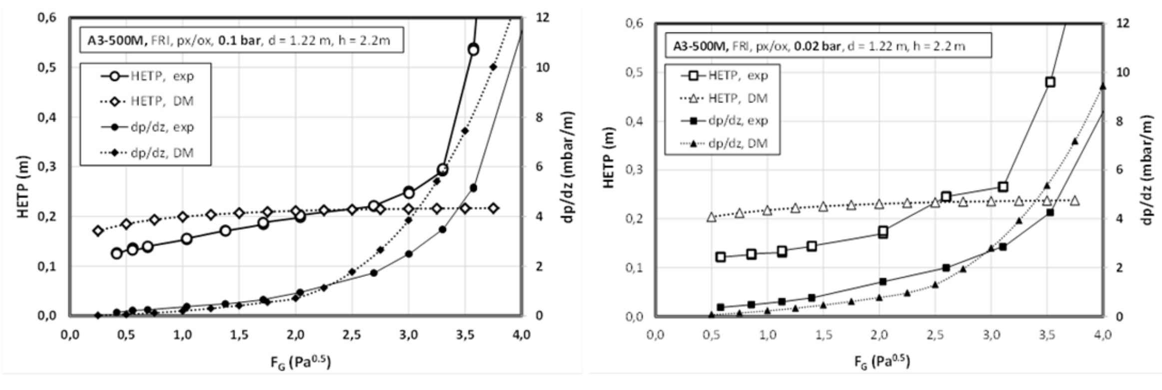

Figure 3: Comparison of measured and predicted efficiency and pressure drop at 0.1 (a) and 0.02 bara (b).

Interestingly, within loading region DM approaches closely both qualitatively and quantitatively measured efficiencies being on somewhat optimistic side. With inertial forces controlling the hydrodynamics, wetted area enhancing capillary effect disappears, and the liquid film driven by gravity tends to flow down at an effective angle substantially larger than the corrugation inclination angle, which is 60^o in present case. Reduction in wetted area and/or contact time causes a strong deterioration in packing efficiency with increasing vapor load, which disappears upon reaching the flood point, which is not accounted for by the model.

Unlike the mass transfer model, the pressure drop model accounts for fluid-dynamic complexities within loading region, using an empirical correction term that becomes active upon exceeding the vapor load corresponding to the point of onset of loading (Verschoof et al, 1999). Strikingly, at 0.1 bara the predicted values match perfectly the measured pressure drop within preloading region, while the predicted pressure drop at 0.02 bara is slightly larger, but the discrepancy with respect to measured curve indicating in this case a considerably higher pressure drop is pronounced. If we follow the predicted pressure drop curves we see that the situation changes upon entering the loading region, where similar to experiment the predicted 0.1 bara curve crosses that of 0.02 bara and exhibits a steeper increase in pressure drop with increasing vapor load. The change in the slope of pressure drop curves corresponds with the onset of loading, which depends on the specific liquid load and therefore, according to DM, occurs at 0.1 bara at a lower vapor load (2.1 $Pa^{0.5}$) than at 0.02 bara (2.32 $Pa^{0.5}$). Measured pressure drop curves suggest that loading occurs in both cases at somewhat larger vapor loads. Thus, in this respect predictions appear to be conservative, but suggest that vapor load corresponding to maximum useful capacity, i.e. flooding limit, should be some 10% higher at 0.02 bara than at 0.1 bara. This, though expected, i.e. complying with all previous experimental experiences, is apparently in conflict with experiment. Namely, the latter indicates that flooding limit is reached earlier at a lower operating pressure. Without adequate experimental confirmation, we may consider this for time being as an observed, useful capacity related “anomalous result”.

On the other hand, operation at 0.02 bara induces fluid-dynamic conditions that could justify an increased efficiency accompanied by an increased pressure drop within preloading region, where film flow prevails. Namely, at such a low operating pressure, a relatively small volume of liquid (specific liquid load < 1 m^3/m^2h) is spread widely by strongly acting capillary forces, ensuring thorough wetting of exposed surface area, and the established film thickness is order of magnitude smaller than the natural roughness of a wire gauze sheet (wire thickness around 0.35 mm). This, a very thin, slowly moving liquid film, in conjunction with surface roughness induced enhancement of interfacial friction exhibited by vapor ascending at a rather high effective velocity (up to 10 m/s), may be considered favorable for mass transfer efficiency. Indeed, additional pressure drop with respect to that experienced at 0.1 bara, observed within preloading region, can be considered as useful one in terms of mass transfer.

Note that DM accounts explicitly for interface roughness effect on the mass transfer (Fair et al., 2000, Olujić et al., 2004). Namely, turbulent flow mass transfer coefficient expression includes the friction factor, and the latter is expressed as function of Reynolds number and the relative roughness, i.e. the ratio of film thickness and hydraulic diameter of vapour flow channel. Since in DM the interface roughness is represented by the film thickness a reduction in film thickness reduces the effects of roughness. Therefore, DM cannot see the roughness of characteristic packing surface texture that is at low liquid loads much larger in the case of a wire gauze packing than the film thickness.

Summarizing, the extent of pressure drop under-prediction by DM experienced in both cases, i.e. at 0.1 bara and particularly at 0.02 bara, within preloading region, is a point of concern. To improve accuracy in this respect, an ongoing research effort is concerned with evaluation of possibilities for a physically sound extension of the present pressure drop model. Owing to the fact that at lower end of vapor loads employed in FRI total reflux distillation experiments the vapor ascends through a sparsely irrigated bed under laminar flow conditions, incorporation of laminar friction into present model appears to be a most promising step in this direction.

4. Concluding Remarks

Total reflux distillation experiments have been carried out with Montz-Pak A3-500M, using FRI low pressure column facility adapted to allow operation at deep vacuum conditions. In addition to standard 0.1 bara test with paraxylene/orthoxylene system, a dedicated test run was conducted at 0.02 bara.

As expected, at both test pressures the best efficiency was achieved at lowest vapor loads, confirming that this advanced wire gauze packing in conjunction with chosen liquid distributor performs exceptionally well at extremely low specific liquid loads (≤1 m^3/m^2h). Since A3-500M packing achieves same efficiency as its conventional counter-part but at a largely reduced pressure drop (Olujić et al., 2013) it is a natural candidate for demanding deep vacuum applications.

Within preloading region the packing exhibited significantly better efficiency at the expense of somewhat increased pressure drop and, strikingly, no gain on capacity side at 0.02 bara.

Delft model proved capable of representing efficiency and pressure drop trends at both pressures. It approaches closely the measured values at 0.1 bara, but largely over-predicts the efficiency and underpredicts the pressure drop within preloading region at 0.02 bara, suggesting that flooding limit at this pressure should be higher than at 0.1 bara.

An ongoing research effort is concerned with finding a physically sound way to reduce or eliminate the amount of under-prediction of pressure drop experienced within preloading region.

A physically sound explanation has been provided for increased packing efficiency within preloading region observed at 0.02 bara, but without adequate validation/reference its plausibility is questionable. Most importantly, FRI should attend properly to observed, capacity related anomaly. Indeed, further total reflux experiments including other large specific area wire gauze and sheet metal structured packings are needed to get certainty about the reliability and accuracy of test results obtained at 0.02 bara using such a large test unit as employed at FRI.

Acknowledgments

We are thankful to Dr. Tony Cai and FRI technical staff for their devoted approach to preparation and execution of total reflux experiments described in this paper.

Nomenclature

a_e	effective (interfacial) area, m^2/m^3
dp/dz	specific pressure drop, mbar/m
F_G	vapor load or F-factor, $Pa^{0.5}$
$HETP$	height equivalent to a theoretical plate, m
HTU_{Go}	height of overall transfer unit, m
k_{Go}	overall mass transfer coefficient, m/s
u_{Gs}	superficial vapor (gas) velocity, m/s
u_{Ls}	superficial liquid velocity, m/s or m^3/m^2h

Acronyms

BTS	Bayer Technical Services
CB/EB	chlorobenzene/ethylbenzene
DM	Delft model
FRI	Fractionation Research Inc.
px/ox	paraxylene/orthoxylene

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