

## The Maldistribution Story - An Industrial Perspective

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For packed distillation columns, maldistribution is by far the most important factor that has a negative influence on performance, especially efficiency. The amount of maldistribution related publications is enormous. For the maldistribution story the research work of the last 50 years is reviewed with a strong focus on structured packing and industrial relevance. The huge effort by packing manufacturers to develop even better products and to establish safe design rules is appreciated, as well as the important developments and support of Fractionation Research Institute (FRI) on this subject.

### 1. Introduction

For packed distillation columns, maldistribution is by far the most important factor that has a negative influence on performance, especially efficiency. At the 1964 meeting of the EFCE Working Party on Distillation, Absorption, Extraction, a major topic was the scale-up of packed columns to larger diameters. The background was that these columns equipped with random packing have been used for about a century, but the efficiency obtained in small diameter columns could not be realized with larger diameters. In several papers published in the fifties this was attributed to wall flow or other causes of maldistribution, especially of the liquid phase.

In 1964 Sulzer started to market its structured packings, made from wire gauze. Their very low-pressure drop was a great advantage for applications in vacuum distillation. Combined with carefully designed internals the column diameter grew steadily, and the efficiency could be kept. The importance of maldistribution has been well recognized and one of Sulzer's major investments was the construction of the 1m diameter column for distillation tests. It was used to demonstrate that the efficiency of structured packing was almost independent of the diameter unlike the behaviour of random packing. With the success of the packings made of metal sheets like Mellapak and others the column diameter stepped suddenly up to 10m and more.

The amount of publications on maldistribution research of the last 50 years is enormous. This paper reviews work involving mainly structured packings with a strong focus on industrial relevance. This approach helps to formulate the maldistribution story. The paper is structured such, that the main achievements are grouped per decade.

### 2. The Fifties

Of course, all research work based on random packing. Structured packing entered the market from 1964 on. But never the less, Sulzer worked on developing the Kuhn column and was heavily fighting against maldistribution problems.

An important theoretical study has been published by Mullin (1957). He used the 2-column model to calculate the effect of maldistribution on efficiency. He assumed a linear relation for the vapour-liquid equilibrium and a constant stripping factor. This simplification allowed an analytical treatment of the efficiency calculation. The theory was then tested with published distillation data from a 125mm column fitted with 12mm Raschig rings. Wall flow was assumed to be the main cause of maldistribution in such small columns and the model was able to deliver a reasonable explanation.

Manning and Cannon (1957) made a theoretical analysis of maldistribution based on channeling. They assumed that a percentage of liquid flows through the bed without mass transfer. The rest of the liquid runs under partial reflux. The apparent number of theoretical stages (NTS) was calculated and served as a

measure of the influence of maldistribution. They concluded that channeling reduces efficiency drastically for very high beds, therefore the use of redistribution devices (interdistributor screens) was proposed.

Another interesting paper originated from Changez and Sawistowski (1963). They improved the theoretical model of Mullin and performed tests with a 2-column arrangement consisting of two 25mm glass-tubes containing 300mm of 3mm single turn Fenske helices. The setup allowed controlled maldistribution and the results agreed quite well with Mullins theory.

### 3. The Sixties

Huber and Hiltbrunner (1966) extended the theoretical analysis further. They used the 2-column-model to investigate the influence of lateral mixing of a maldistributed column on the performance.

Two cases were distinguished: continuous lateral mixing in the vapour phase in a single column or stage wise partial mixing of the rising vapours between 2 partial columns. They found that it does not make a difference. Packings with inherent strong lateral mixing depend less on maldistribution and frequent redistribution of the liquid phase helps to decrease the influence of maldistribution.

Meier und Huber (1967) tested the theory of Huber and Hiltbrunner experimentally with artificial maldistribution in a test column of 0.5m diameter with 2.67m Sulzer gauze packing BX. The reflux distributor had 184 drip holes, which have been plugged by 10, 30 and 50% on one half. The maldistribution parameter  $\lambda$  resulted in 0.05, 0.176 and 0.33 according to Sulzer's definition:

$\lambda = (L_1 - L_2) / (L_1 + L_2)$ , with  $L_1, L_2$  the liquid streams in the 2 partial columns.

Meier und Huber, 1969, stated that "a packing suitable for industrial application must give the same efficiency independent of column diameter". Their paper is a summary of several activities, Sulzer undertook to prove that structured packing efficiency does not depend on diameter. The efficiency of BX was almost the same when measured in four different columns of 70, 250, 500 and 1000mm diameter (see figure 8 in their paper). The tests were done at 133mbar top pressure, trans-/cis-decalin as test mixture under total reflux.

This was very important because the diameter of the industrial columns where these gauze packing BX and CY were applied grew steadily. The authors mentioned already diameters up to 2m.

The other interesting information was that Sulzer had a 1m column for distillation tests at their disposal since 1966. The column was also used to perform FRI tests for gauze packing in 1971.

### 4. The Seventies

The Sulzer gauze packing was applied in a steadily growing number of application. Its main domain was the vacuum distillation because of the extremely low pressure drop. Using pipe distributors with a high number of drip points, the packing was uniformly irrigated on top and due to the self-wetting properties of the gauze no maldistribution was generated. The design was done very conservative and packing heights did not exceed 4m as a rule.

At the AICHE 1976 Sulzer presented Mellapak, a further addition to the structured packing family. It consisted of corrugated sheet metal with a well-defined surface structure. Because of the drastically lower cost it became a real success. The diameter of the columns grew steadily. An important application was the separation of ethyl benzene/styrene. The typical case was a revamp from trays to Mellapak. And now maldistribution became an issue. The challenge was to design the liquid distributors to deliver the liquid evenly over the cross-section. The common type was a so-called element type, consisting of parallel arm channels which were liquid supplied by means of one or more main channels above.

The academic world was still busy in investigation of the behaviour of liquid flow in random packings. Many papers have been published, among others the article by Bemer and Zuiderweg (1978) may be consulted for more information.

### 5. The Eighties

The market for structured packings developed rapidly. Very large columns have been revamped from trays to structured packing, among others styrene and caprolactam towers for the chemical industry. The refineries discovered the advantage of low pressure drop as well, especially for their vacuum towers.

Research on maldistribution was resumed in the eighties both in academia and industry.

Sulzer started further work on maldistribution. It was the time when China was recovering from the culture revolution. Mr. Yuan from Tianjing university spent more than a year with Sulzer analysing with an in-house developed computer program the impact of maldistribution on packing efficiency. The numerical work was afterwards supported by experiments in Sulzer's 1m test column (Yuan and Spiegel, 1982). The packing used was Sulzer gauze packing BX with a height of 4m operating at a top pressure of 50 mbar. Trans- / cis-Decalin was the test mixture. To generate maldistribution some drip holes of the distributor were blocked on one half

of the cross section. The maldistribution parameter varied between 0 and 0.20. Partial reflux runs were done as well. For a safe design a limitation of the bed height of 15 to 20 theoretical stages was recommended.

A very important study was done by Hoek (1983) at the university of Delft. He measured the liquid distribution at the bottom of a packed bed with 681 measuring points. The column had a diameter of 500mm, test fluid was water, no counter current of air. He varied the height of the bed in discrete steps to obtain the axial development of the flow profile. Following packing were used: Mellapak 250.Y, 500.Y, BXPFP (plastic gauze packing). The work proofed the existence of a small-scale maldistribution in structured packing and the existence of wall-flow as well, but small as compared to the one in random packing. A comprehensive summary can be found in Hoek et al. (1986).

His work inspired other researchers at Delft university for further investigations. Among others Stikkelman (1989), who introduced a counter-current flow of air.

The trend to ever increasing column diameters put the need of qualifying liquid distributor more and more in the centre of design questions. Sulzer decided to build a large test rig for distributors up to 10m in diameter operated with water. It was capable to measure the liquid flow from individual or a group of drip points (area measurements). For trough type distributors the standard test consisted in measuring the liquid level in the arm channel and to calculate the coefficient of variation (COV) as a quality measure.

A COV of less than 4% was labelled very good, less than 7% good. With all the information gathered in the late eighties a standardisation of the channel dimensions was done that allowed to guarantee the distribution quality as well as the economic use of material.

Sulzer thought that area measurements provided much better information of the distribution quality than measuring the variance of the outlet from each drip point. However, manual area measurements were very time consuming. Therefore, with the availability of low cost computers the process of positioning the collecting devices beneath the distributor could be automated. Area measurements proved to be extremely valuable for the evaluation of predistributor quality.

The use of COV as a quality feature of liquid distributors has been discussed by numerous authors. It cannot account for the scale of maldistribution, but only on its degree. This is nicely illustrated by Zuiderweg et al. (1993) for four maldistribution cases having approximately the same COV but different scale. Moore and Rukovena (1987) developed an empirical quality index, however applicable mainly for random packings.

Publications relating to the design of large diameter distributors are scarce. Albert and Tokerud, 1995, emphasised the importance of the many mechanical details that have to be considered beside the process related issues.

## 6. The Nineties

Structured packings were booming in the Chemical industry as well as in the oil and gas industry. Another interesting application was air separation that required a minimum volume for the internals in the cold-box. Therefore, packings with high interfacial area were preferred. It was already known that beds with many separation stages were sensitive to maldistribution. Important air production companies started their own confidential inhouse investigations. An example of such work can be found in Kalbassi and Zone, 2002. Air Products built a cryogenic demonstration unit with an internal diameter of 900mm. Tests were run in a short and a long bed with a packing of interfacial area of  $500 \text{ m}^2/\text{m}^3$  using  $\text{Ar}/\text{O}_2$  as test mixture. No information was given about the actual bed heights and the distributor type. The composition distribution was measured below the bed within 5 zones at total reflux. The COV for the short bed was calculated to 0.018, and 0.032 for the longer bed, a clear indication that maldistribution increases within the packing.

Another interesting project started at Fractionation Research Institute in Stillwater, Oklahoma (FRI). They performed studies with controlled liquid distribution using Pall rings as test packing. The suggestion came from Zuiderweg, who worked as a consultant. He noticed that the efficiencies for Pall rings measured by FRI were considerably lower than those measured by Billet (1967) in a 800mm column. Zuiderweg supposed that the cause of the differences had to be sought in the distributor. FRI used the v-notched trough distributor with a much lower distribution density than the distributor used by Billet. Therefore, they fabricated a tubed drip pan distributor with 100 drip points. The efficiencies for 25mm Pall rings increased by 50% (Kunesh et al., 1987). FRI continued the project by fabricating an adjustable liquid distributor (ALD) and performed several tests with typical forms of maldistribution patterns found in industrial columns. One major result was that large-scale maldistribution in form of zones with high and low liquid flow had a dramatic influence on efficiency.

For larger columns the packing had to be divided in segments. These segments had to be assembled to build a coherent layer across the cross-section. But between the segments there were always thin gaps. Stoter (1993) picked up concerns from industry regarding the influence of segmentation gaps on the formation of maldistribution within structured packing. He used a rectangular simulator, 3m long and 0.5m wide, with air/water in counter-current. The device was divided into 15 compartments to measure the liquid and gas

distribution. Packings were Ralupak 250.YC und Montz Pak B1 250, both segmented, packed heights were 2.2m and 4m. He was able to measure the influence of the gaps between the segments on the flow distribution.

Zuiderweg et al. (1993) introduced the zone/stage model to predict the spatial distribution of the liquid flow in a simplified structure: the column is decomposed in horizontal direction into zones (annular, segmental, sectoral) and in vertical direction into stages of height HETP (Height Equivalent to Theoretical Plate). The wall region is considered only for the annular case, not for the others. The liquid leaving a cell is split to flow to different cells in the layer beneath, according to the value of lateral spreading. The gas phase flow profile is uniform, but lateral mixing is assumed. Once the fluid streams for each cell are established, mass transfer in the cell is calculated. Because the height of the cell is one HETP, leaving streams are in equilibrium. The model allowed to calculate qualitatively the effect of a large-scale maldistribution on efficiency.

Edwards et al. (1999) discussed the value of the coefficient of variation (COV) as a measure of maldistribution. They argued that it provides useful insight into the level of maldistribution, but it is not a good indicator of the depth of penetration of the maldistribution into the packed bed and its impact on efficiency. They developed a theoretical method to calculate the depth of penetration for the liquid and the vapour phase separately. They could show that maldistribution in the vapour phase is equalized much faster than in the liquid phase. This is confirmed by tests of FRI in the 1.2m column (Cai et al., 2003). Therefore, maldistribution in the vapour phase is of concern only in shallow beds of large diameter. Regarding maldistribution in the liquid phase the penetration depth was calculated for the cases used by Zuiderweg et al. (1993). It became clear that the penetration depth depends strongly on the scale of maldistribution.

## 7. The 2000ies

This decade was characterized by the success of the second generation of structured packings, the high capacity packings: MellapakPlus and Flexipac HC already at the end of the nineties, Montz Pak M/MN series and Raschig Superpak a few years later. Also, there was an amazing innovation with respect to liquid distributors and vapour inlets.

Sulzer wanted to clarify the open questions regarding redistribution. Do liquid streams have to be totally remixed? Or is it enough to restore a uniform distribution (hydraulic equalisation)? In cooperation with the university of Dresden a project was started. The subject was treated theoretically and experimentally by Bartlok (2003). The theoretical part consisted of numerous simulations with a modified 2-column model using PRO/2. The model consisted of a network of 4 columns which were pairwise arranged to build a top and a bottom section. The connecting streams could be adjusted to simulate total or partial remixing of the streams between the top and bottom section. With side streams from each stage vapour or liquid mixing within the sections could be simulated. The reflux to the top section is unevenly distributed to the two columns to simulate maldistribution. The distribution of the reflux to the bottom section is either totally mixed (concentration) or partially mixed, but the liquid is evenly distributed to the two beds. The simulation showed that there is only a small difference in the efficiency of the lower bed for the two cases, as long as the section height is kept below 20 theoretical stages which is in line with the well-known design guidelines.

Experiments were done in Sulzer's 1m column. Two beds of packing Mellapak 752.Y were installed with a special redistribution device between. This device was fabricated such that the liquid streams from above could be mixed or not. The mixing unit of the device was put outside the column and allowed various setups. Artificial maldistribution was generated by the top distributor by plugging a number of drip holes on one half of the cross-section with a range of maldistribution parameters from zero to 0.45. The experiments confirmed the results of the simulation.

Olujić et al. (2006) started air/water experiments to compare the liquid distribution quality of a high capacity structured packing (Montz Pak B1-250M) with its conventional counterpart (Montz Pak B1-250). The experimental program comprised 3 different bed heights and various combinations of liquid and gas loads in an air/water simulator of 1.4m internal diameter. They found that the distribution quality for both packing was comparable, but the high capacity packing tended to somewhat higher wall flow. Olujić and Jansen, 2015, published additional measurements performed in the same simulator with various drip point densities and artificial maldistribution. The experiments were thought to be an extension of those done in the FRI study (Fitz et al., 1999). They showed that a reduced drip point density ( $50/m^2$ ) has no negative effect on COV as compared to a normal drip point density ( $100/m^2$ ). Another test involved severe initial maldistribution in form of chordal, peripheral and central blanking of the drip holes by approximately 50%. It has been shown that the packing could not restore to uniform distribution. Consequently, it is absolutely necessary to start with a uniform initial distribution on top of the packing.

## 8. The 2010ies

The use of structured high capacity packings is now well established. Due to their higher capacity at comparable pressure drop they allow to revamp columns with conventional structured packings. There are only two large manufacturers left (Sulzer and Koch-Glitsch) who own an enormous know-how. Maldistribution caused by liquid distributors seems to be not a big issue anymore. But there are still other sources that must be considered carefully. Kister's book (2010) contains many illustrative examples of possible failures.

Academic work on maldistribution continues. For example, at the Institute of Thermophysics in Novosibirsk efficiency measurements with Flexipac 1Y in a distillation column of 0.9m internal diameter using a Freon mixture have been done (Pavlenko et al., 2006). Interestingly, a large-scale maldistribution established within the packing despite a uniform initial distribution. After numerous tests looking for the cause they detected that the vapour phase was unstable due to an inverse density gradient (negative stratification), see Pavlenko et al., 2010.

At the Technical University of Munich another study on maldistribution has been started (Hanusch et al., 2017). An air/water simulator with 1.2m internal diameter is used to investigate the liquid flow distribution in beds of modern random packing like Raflux Rings. The packed height varied from 1 to 3m. Below the packing the liquid flow density is measured in total 19 elements (6 annular segments, divided into sectors). The experiments aim to gather spreading coefficients to be used for validation of the cell model. It is hoped that the study is extended to structured packings as well.

## 9. Conclusion

The basic rules for a maldistribution tolerant column design are well known and established. Nevertheless, errors in design, construction and installation may always occur. Important is a sensitisation for the subject and a sufficient treatment in education. Most of the academic work has been done in small scale devices with the focus on the flow pattern within the packings. Industry has been more interested in how to prevent maldistribution. Therefore, a main topic has been and still is an optimum design of the liquid distributors and redistribution devices.

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