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STRUCTURED PACKING EFFICIENCY – VITAL INFORMATION FOR THE CHEMICAL INDUSTRY

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Abstract
Distillation is the most important thermal separation process, with separation efficiency as one of the fundamental parameters to influence economy and energy consumption. The precise knowledge of separation efficiency and the ability to compare different types of column internals is vital information for the chemical industry. Still, experimental data is the only significant source of such data. The authors have taken the effort to work out in detail different factors influencing both measurement and interpretation of structured packing separation efficiency data, to provide basis for establishing an open standard in this respect.

Keywords: Distillation, efficiency measurement, standardization

1. Introduction

Distillation is the most important thermal separation process. The trends in distillation are going for a reduction of energy consumption and an increase in column size and complexity, including hybrid processes. A strong competition for economic advantages in a highly dynamic market can be observed, both on the side of vendors and manufacturers of distillation columns and equipment, and on the user side, e.g. the chemical and related industries. Separation efficiency is one of the most important factors in operation and design of distillation columns; it is one of the fundamental parameters that influence economy and energy consumption. The precise knowledge of separation efficiency and the ability to compare different types of column internals offer an economic advantage for both users and vendors.

Accepting the huge advances in the past decades with respect to the creation of predictive models that help in the design of distillation columns, experimental data is still the only significant source for column internals development as well as for model validation. Research and development and thus efficiency measurements are done at universities, various institutions, manufacturers, and by end users, worldwide. Depending on the means available and according to local customs, measurements are done following quite different standards. They differ in pressure, test systems, experimental equipment to name just a few. Moreover, the measurement method details are not always transparent resulting in considerable uncertainty affecting the reliability of the data. Unfortunately, as a result these data are often not comparable. But not only the data generation is itself affected. As raw data is published only rarely, the analysis of the data comes into focus. Again, evaluation of the data is done following different standards. Shortcut methods are used frequently using different assumptions. Such assumptions may or may not be valid depending on the measurements that have been made. Depending on the physical properties used for evaluation and on the type of data reduction and reconciliation, the same data may result in quite different apparent separation efficiencies. Moreover, a detailed error analysis is seldom made. These inconsistencies in the evaluation and interpretation of the measurements make the use for column design difficult.

1.1 A standard for efficiency measurements

The implementation of a common standard making efficiency measurements comparable is highly desirable, for equipment manufacturers, industrial end users as well as academia. Efficiency in this context can take two different aspects, which are relative comparability and absolute values. While relative comparability is needed for a fair comparison between different categories, types and producers and is a prerequisite for the correct choice of equipment, reliable absolute values are needed for scale-up, column design, and realistic device rating. The two aspects have different requirements regarding the efficiency measurement and data evaluation, which will be discussed later on.

In a previous effort, the authors have worked out different factors influencing both measurement and interpretation of structured packing separation efficiency data, to provide a basis for establishing an open standard in this respect. An overview of the state of the art has already been made publicly available[1]. The presentation gives an overview with emphasis on recent developments and amendments in this process. It highlights the necessity for establishing a standard for distillation separation efficiency tests, which will enable realistic evaluation and comparison of performance characteristics of structured packing and facilitate the scale-up and tight (re)design of distillation columns in industrial practice. Tangible suggestions are made as to what this standard could contain.

2. Best practices in efficiency measurement

2.1 Measurement apparatus and procedure

A comprehensive overview of equipment and measurement procedures commonly used by established institutions and companies is given elsewhere[1]. This knowledge may well serve as a starting point for deducing best practices. Essential measurements for determination of separation efficiencies are: absolute pressure, pressure drop over the bed to be rated, temperatures in the column, reboiler and condenser, compositions above and below the bed, reflux flow rate and energy balance. Necessary operating top pressures are 100 mbar and 960 mbar, which cover the typical range of application for structured packing. Higher, lower, and intermediate pressures can be added according to the type of application. Usually, an intermediate pressure of 330 mbar is taken as well. Pressure drop measurements not only over the entire bed but also over separate sections can give useful information on flooding, and can be used for data reconciliation. Typical pitfalls are the pressure gauge lines, which have to be sized and installed carefully to avoid plugging by vapor condensing into the lines. Likewise, the temperatures above and below the bed are necessary for consistency checks of the measurement. An additional temperature column profile can be very useful for evaluation. Adding several temperature sensors in a cross section can be used to judge on maldistribution. A concern in this respect is the degree of allowable subcooling of the reflux, which should be kept low enough to avoid uncontrolled variations of liquid and vapor flow rates in the bed.

It should be ensured that concentration measurements are single phase, which is not always trivial. The concentrations should be kept in a range where analysis, e.g. by gas chromatography, can produce reliable results. Extreme purities should be avoided. Taking additional measurements that would allow establishing a composition column profile is highly desirable. However such measurements are demanding. The major concern is that representative samples are drawn, which contain liquid or gas phase only and give a good average of the cross-sectional concentration. Samples taken from the sump may be helpful as well. However, depending on column geometry and reboiler type they will differ from the below-bed sample and cannot be used as a replacement.

As good wetting of the surface is mandatory to achieve good separation efficiencies, proper design of the liquid distributor in the test facility is a primary concern. It is desirable for reasons of practicability and comparability that the distributor be not changed during the tests. This requires a high turndown, high free-area trough distributor. A turndown ratio of 10 is sufficient for most applications; at least 60% free area being a good measure. Drip tubes which extend below the bottom of the trough are preferred because they allow higher gas velocities / F-factors than other types before liquid entrainment occurs due to the constriction of the gas flow. A drip point density of 150 / m^2 is considered sufficient for most applications, namely the testing of packing with a specific surface area of at least up to 500 m^2/m^3. Initial vapor distribution is a minor concern. However the distance between packing support and vapor inlet should be maximized and, preferably, a liquid collecting/vapor redistributing device should be installed at least one column diameter below the bed, which, importantly, also allows extraction of a representative liquid sample.

To obtain significant efficiency measurements, a diameter of the column of 0.4 m is recommended. This value originates from the consideration that twice the element height of the structured packing should be the minimum. Smaller column diameters will result in measurements which are dominated by wall effects. Bigger diameters will make equipment and operation more expensive, so that it seems reasonable to choose the smallest acceptable diameter. This is especially true for the objective of relative comparability of equipment. The transfer to other diameters is in the scale-up knowledge of the engineer.

Figure 1: Installation of a new column for efficiency measurements at BASF, Ludwigshafen. The column body can be pivoted for easier installation of internals.

The column should be large enough to contain approximately 20 theoretical stages, which is equivalent to an installable bed height of 4 to 6 m. It is well known that shorter beds perform better, as the liquid distribution tends to deteriorate with increasing bed height. This is considered a geometry dependent feature of the packing, which makes it difficult to predict efficiencies for other bed heights, as analogies are difficult. As installations of structured packing in manufacturing plants nowadays usually use bed heights of 15 – 20 theoretical stages, efficiencies measured at smaller bed heights need to be applied with care as they might be too optimistic.

It is obvious, that the installation of the internal to be tested should be done according to industrial standards. The proper configuration of wall wipers and the rotation of the elements and the liquid distributor are beyond the scope of this article (see reference[1]).

It is good practice to document geometry details like corrugation and crimp angles, element height and perforation. This helps in keeping track with the ongoing development of internals. The surface structure should be quantified as well, if possible, as it does add to the specific surface of the packing and influence the distribution of liquid on that surface. Those effects influence the effective mass transfer area and thus the separation efficiency.

2.2 Test systems

Various test systems have been considered suitable for measurement of separation efficiency. Those recommended by the EFCE Working Party “Distillation, Absorption and Extraction”, presently “Fluid Separations”, can be found in a booklet compiled by Onken and Arlt[2]. Predominantly in use out of this selection are chlorobenzene/ethylbenzene (CB/EB), orto-/para-xylene and cyclohexane/n-heptane (C6/C7), which appeared suitable for testing structured packing under vacuum and near atmospheric

pressures. However, it should be mentioned that even measurements with these recommended test systems will produce different results. This is due to the physical properties of the systems. Both aspects of comparability – relative and absolute – are affected. This rather unpleasant situation can only be amended by further reducing the number of test systems, preferably down to one.

To illustrate criteria how to find the most preferred test system from those mentioned above, the widely used C6/C7 system is reconsidered. It exhibits a concentration dependent slope of the equilibrium line. The relative volatility varies between 1.62 and 1.76 over the concentration range at ambient pressure. As the relative volatility goes directly into the calculation of the separation efficiency, no matter what evaluation method is used the obtained HETP will depend on the concentration range the measurement was made in. The authors have encountered relative deviations of as much as 25% HETP for the same packing measured under otherwise identical conditions by the same experienced staff. Deviations of 15% can be considered the rule rather than the exception.

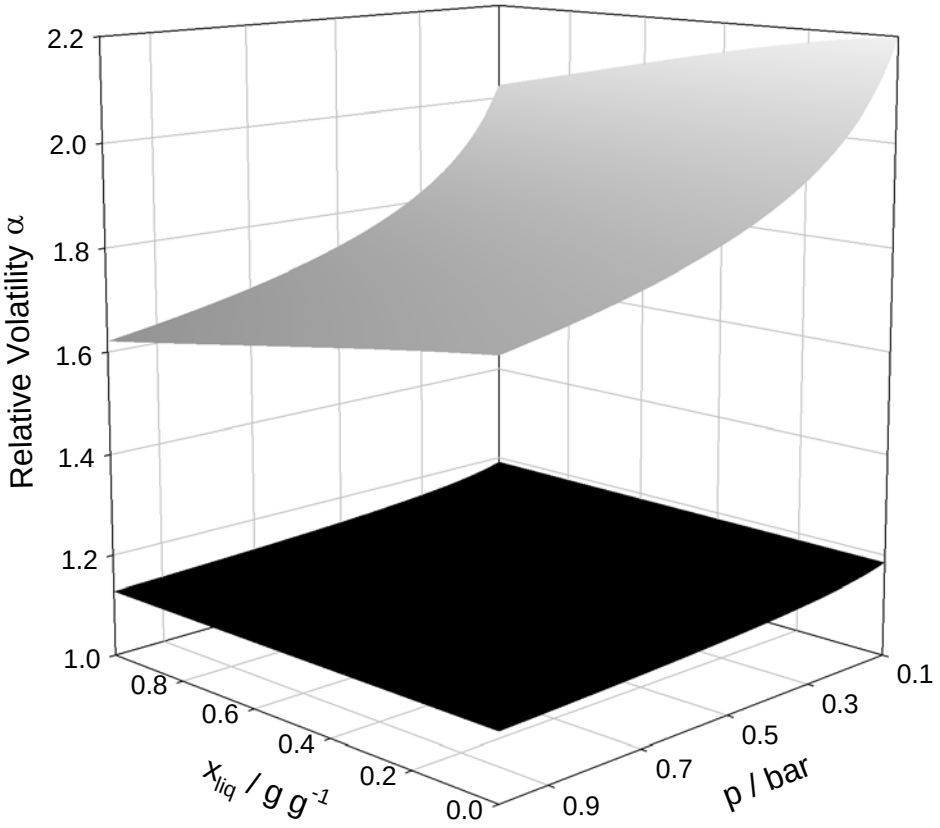

Figure 2: Relative volatility of C6/C7 (light gray) and CB/EB (black) over pressure and concentration range. It demonstrates the strong dependence of C6/C7 and the constant behaviour of CB/EB.

Such uncertainties are certainly not acceptable from the point of view of contemporary competition – be it manufacturer or end user. This issue is not only problematic with respect to comparability of different packing or with their application. Deviations in that range will easily be larger than improvements achieved during research and development of new packing. To worsen the situation, depending on whether the system has been considered ideal on non-ideal during the evaluation of the concentration data, the relative volatility will be higher at one end of the concentration range or the other. To avoid such thermodynamic uncertainties, the authors recommend the use of CB/EB. This system is slightly lighter than orto- / para-xylene and exhibits a slightly lower relative volatility, constant over the entire concentration range for a given pressure or temperature. Due to its rather low relative volatility, it is suitable for measurement of high theoretical stage numbers, which is desirable as explained above.

It has been criticized, that the liquid densities of chlorobenzene and ethylbenzene do not run perfectly in parallel over the temperature and pressure range of interest. This is only an issue if results are to be visualized over the c-factor rather than the F-factor, as a proper description of the liquid density is required. This is however not a fundamental problem like the relative volatility issue mentioned above.

3. Data evaluation

3.1 Shortcut methods

HETP (height equivalent of a theoretical plate), being the most common and most practical measure for separation efficiency, is usually calculated by the Fenske equation. Fenske[3] made the assumptions of total reflux, constant relative volatility and constant molar overflow. The former being the standard and recommended mode of operation for efficiency measurements, the latter two depend on the thermodynamic properties of the test system. Its sensitivity to errors has been investigated many times. It can be concluded e.g. from Deibele and Brandt[4], that the error is usually low provided the assumptions are met, the concentration range of the measurement is restricted, the number of stages is properly chosen, and good parameters have been used for evaluation, e.g. for the relative volatility. In the pressure range mentioned above, an error of 1% can be expected from uncertainties in available (measured) relative volatility data. Additional errors (<0.5%) will have to be expected if the concentrations during measurement are limited to the range 9 mol% to 91 mol%, and a theoretical stage number around 20 is chosen[5]. Those values are valid for the CB/EB system, for other systems the numbers look different. In any case, data evaluation should include an error propagation analysis.

As the physical properties of the test mixture will change over the height of the column due to changes in composition, flow and pressure, a representative state for comparison has to be chosen. It is recommended to use mid-bed conditions, although the temptation exists to use best-case or worst-case data, which are usually found above or below the bed. If no better sources for estimates are available, arithmetic averages for capacity related quantities like F-factor and a geometric average for the relative volatility are recommended. Figure 3 gives an example of the influence different operating conditions can have on the evaluation of a single dataset. As the measurements for this example were made with the C6/C7-system, it demonstrates also quite well the influence of the change in relative volatility over the height of the column. It has to be noted that Figure 3 is based on a single measurement, i.e. one concentration difference per data point. As mentioned above, measurements with the C6/C7 system in different concentration ranges will additionally result in differing HETP values.

3.2 Rigorous evaluation methods

Rigorous evaluation methods are encouraged, as they circumvent some of the limitations mentioned above. With the advances in information technologies, namely the pervasiveness of – in relation to the problem – unlimited computing power and the availability of suitable software packages, the effort required to make a rigorous calculation for evaluation of measurement data is the same as for shortcut methods.

As HETP is the desired measure, a rigorous (stage-to-stage) calculation seems especially fit for the purpose. No dedicated process simulation package is needed for this purpose, a spreadsheet calculation is sufficient. Heat balance effects like internal reflux generated by subcooled liquid or heat losses due to insufficient insulation are easily accounted for in such a calculation – if they can't be completely canceled out during the measurement itself, which is to be preferred of course.

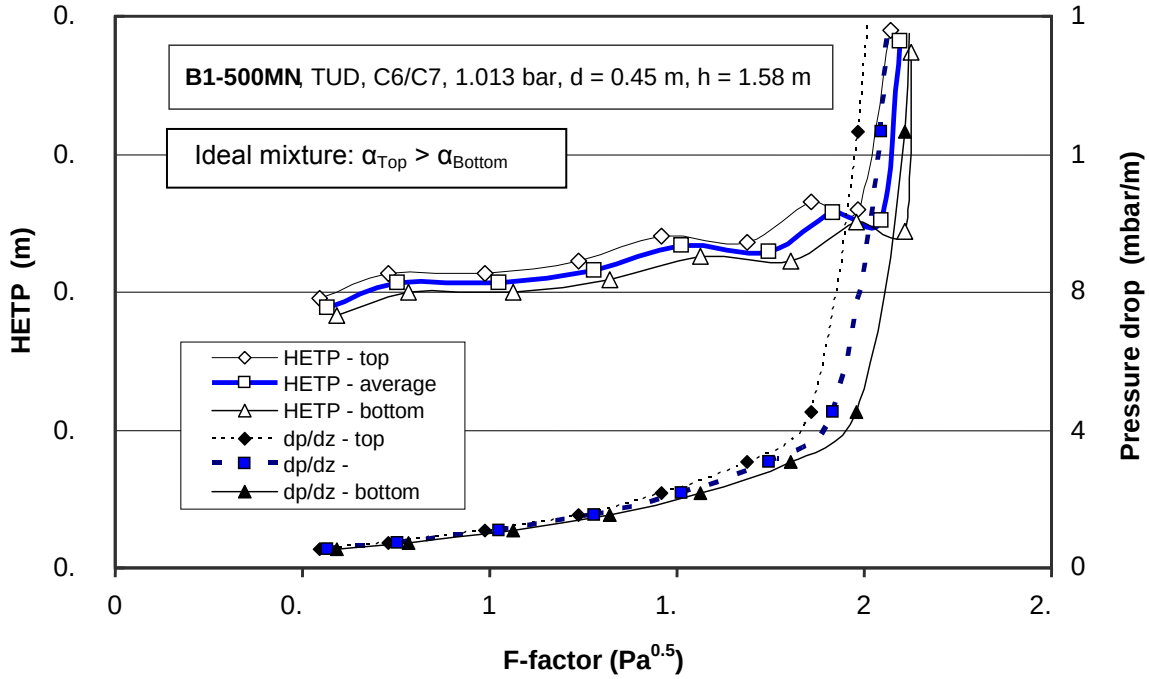

Figure 3: Effect of operating conditions on packing efficiency and pressure drop for the C6/C7 system.

Although the error to be expected from shortcut methods can be in the low single-digit percentage as shown above, a stage-to-stage or piecewise linearized calculation offers the opportunity to further improve accuracy. Intermediate values are calculated automatically and averaging can be reduced, e.g. of the relative volatility. As an additional benefit, some data reconciliation and plausibility checks are possible even with a simple stage-to-stage calculation as e.g. intermediate temperatures are calculated which can be compared to a measured temperature profile.

4. Conclusions

Precise and comparable data on separation efficiency for column internals is vital information for all parts of the chemical industry. It should be a matter of mutual interest for end users, manufacturers and academia. Unfortunately, notably the latter often publish data obtained under fully inappropriate test conditions. The need for a common standard is obvious, as results differing in the double-digit percentage due to the measurement procedure are neither necessary nor desirable. A document describing the state of the art is available[1]. In this presentation the most essential parts of a possible standard have been highlighted: a single test system CB/EB, a large enough column, equipment chosen with the background knowledge provided and suitable experimental and data evaluation procedures.

Although here the emphasis was on structured packing testing under total reflux distillation conditions, the same is generally valid for testing of random packing.

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