Distillation Column Internals/Configurations for Process Intensification*

Ž. Olujić, B. Kaibel, H. Jansen**, T. Rietfort**, E. Zich**, and G. Frey****
TU Delft, Laboratory for Process Equipment,
Leeghwaterstraat 44, NL-2628 CA Delft
**Julius Montz GmbH, Hofstrasse 82, D-40723 Hilden

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In memoriam Prof. Emeritus Franc Šef

The purpose of this paper is to introduce some recently commercialised packed column internals and configurations developed at J. Montz company in close cooperation with universities and industry, which by the virtue of their nature intensify in some way the distillation process. These include state of the art high capacity structured packings, hybrid packed beds with partially flooded sections, streamlined liquid collectors, catalytic packings and the dividing wall column (DWC). The later one, an exclusive development realised in a close cooperation with BASF company, represents a major technological breakthrough; recent advances being mainly reflected in increasing both mechanical and process design flexibility by introducing a number of proprietary designs of DWC components. This paper discusses the backgrounds of developed technologies, the related state of the art and the perspectives for further development.

Keywords:
Packed columns, structured packings, catalytic packings, column internals, divided wall column

Introduction

Certainly the distillation is the most widely applied separation technology and will continue as an important process for the foreseeable future because there is simply no industrially viable alternative around. Also, confronted with challenges from other technologies distillation improves and from time to time breakthroughs are made which move this technology to a higher level of sophistication. Nevertheless, as an energy intensive separation, distillation is under permanent pressure of increasing energy costs, and further developments are needed to preserve its pre-eminent position in the separation field.

Although distillation is generally recognized as one of the best developed chemical processing technologies there are still many technical barriers that could, when overcome, secure the position of the distillation and even make it more attractive for use in the future. A thorough overview of the technical barriers and related research needs can be found in the VISION 2020, 2000 Separations Roadmap published by AIChE in cooperation with the U. S. Department of Energy.[1] Here, the equipment performance was ranked equally as the next highest priority barriers after lack of fundamental data.

Because of the fact that in the past two decades the support for equipment performance related research declined strongly in industry and universities the equipment related developments were left mainly in the hands of manufacturers. Regarding the complexity and the scale of related research and development effort, as well as limited financial resources, this appeared to be a heavy burden for a well-established medium size company such as J. Montz. Being in similar situation regarding the lack of support, the Laboratory for Process Equipment regarded a close cooperation with Montz as a chance to maintain the quantity and quality of fundamental research in the contacting equipment field. Another essential ingredient of this association was a close cooperation of Montz with a group from BASF, around Dr. G. Kaibel, active in improving the packed column technology utilised mainly in fine chemicals applications. In addition, both J. Montz and BASF cooperated closely at specific developments with other universities. Looking back, we may say that working in the triangle equipment manufacturer, industrial user and university enabled cross-fertilization of new ideas and models while protecting the proprietary know-how.

Certainly, J. Montz GmbH is a manufacturer of all kinds of process equipment, however this paper

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Corresponding author: z.olujic@wbmt.tudelft.nl

focuses on recent developments, i.e. process intensification advances in packed column technology, including improvements of packings and internals, development of a catalytic packing, as well as that related to unique knowhow regarding the design and construction of divided wall columns. The purpose of this paper is to present major features of developed technologies, accompanied by a survey of the related state of the art and to indicate the potential for further improvements.

High capacity/efficiency structured packings

Development of internals for small and large scale vacuum distillation applications is a traditional field of activity of J. Montz related strongly to TU Delft. Namely, over the years, J. Montz fully or partly supported the packed column internals related research work at the Laboratory for Process equipment that resulted in four PhD theses.[2–5] Last ten years, the joint effort vent largely into improvement of the performance of established structured packings as well as to the development of an overall predictive model. Most of the performance screening work has been done using hydraulic facilities available at TU Delft and J. Montz in Hilden.[6,7] Pilot scale, total reflux experiments carried out in a systematic way in cooperation with the Separations Research Program (SRP) at the University of Texas at Austin (Dr. A. F. Seibert and Prof. J. R. Fair) provided experimental evidence on the performance of conventional and advanced packings (M series) in the wide range of operating conditions.[8,9] The conventional packings data provided also a basis for evaluation and improvement of the predictive accuracy of SRP and Delft models.[10]

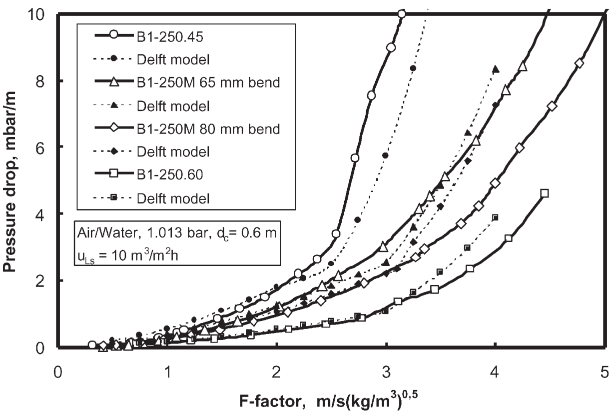
While studying the hydraulic behaviour of conventional structured packing with different corrugation angles it was observed that overall pressure drop comprises three major components: gas liquid interaction at the interface along the flow channels, flow direction change losses with associated entrance effects at the transitions between packing layers and very influential but often ignored one, the gas-gas interaction at the plane separating crossing gas flow channels. The latter two, which are responsible for, say 80 % of total pressure drop in packings with a corrugation angle of 45 degrees, do not contribute greatly to mass transfer. This means that reducing these pressure drop components appropriately could lead to increased capacity without affecting adversely the mass transfer efficiency or vice versa. Both options have been evaluated thoroughly.

In fine chemicals distillations usually high product purity is main concern, and therefore the performance improvement effort is oriented mainly toward improving the efficiency of contacting devices. A possibility for increasing the efficiency of structured packings is discussed first, which implies implementation of a geometry modification that could help to maximize the ratio of "useful" frictional pressure drop to "useless" pressure drop due to gas/gas interaction. By inserting (sandwiching) properly designed flat sheets in between corrugated sheets,[11] both objectives were achieved in one move. Tests were carried out with a prototype based on standard corrugated sheet metal packing of the type B1–250. The large channel, monolith-like structure with substantially enlarged specific surface area produced a lower pressure drop, and consequently allowed a substantial capacity increase. However it appeared to be prone to development of severe liquid maldistribution, which, particularly in preloading region appeared to be highly detrimental to efficiency. Some advantage of this simple modification, which allowed a significant increase in surface area with respect to packings with the same hydraulic diameter, could eventually be expected in applications where capillary forces dominate the spreading of liquid.

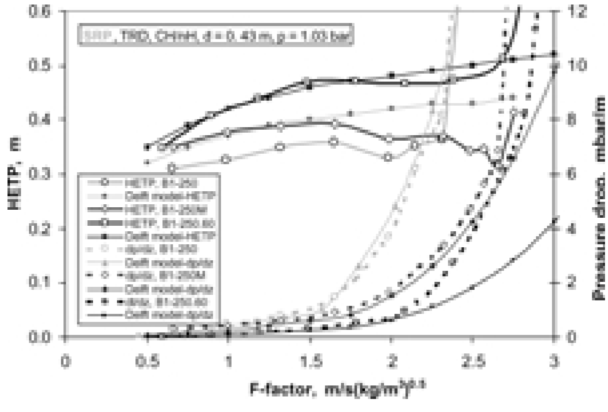
As demonstrated by researchers from the TU Munchen,[12] it appears that the maximal lateral transport per unit height of both gas and liquid, imposed by closed channel structure, could be of some practical use. Namely, two packing elements with such a structure, rotated to each other by 90 degrees proved to work as a short but highly efficient gas and liquid redistribution section for random packings, contributing also to overall mass transfer performance. Such a redistribution section could also work in conjunction with large specific surface area structured packings, however, due to limited extent of radial transport within one packing element, this configuration may eventually be an elegant solution for savings in the height of small diameter columns only.

A real breakthrough was achieved with second, capacity increasing option.[9,13] The essence of this development is the minimization of gas flow pressure drop created at the transition between packing layers, while allowing a smooth drainage of liquid to the layer below. This was achieved simply by bending smoothly the lower part of corrugations from 45 to 90 degrees (see the photograph shown in Figure 1). Measured pressure drop and efficiency curves shown in Figures 2 and 3 clearly indicate that bending only the bottom part of corrugations to the vertical can result in a substantial reduction in pressure drop of conventional structured packing accompanied by a significant increase in the capac-

F i g . 1 – *Photograph of 0.45 m diameter elements of common size B1-series packings with inclination angles of 45 and 60 degrees, respectively, and the bend in the bottom part (B1–250M) of corrugated sheets*



F i g . 2 – *Comparison of pressure drop performances of B1–250, B1–250.60 and B1250M indicating the effect of the corrugation bend length; measured vs. calculated*



F i g . 3 – *Comparison of total reflux distillation performances of B1–250, B1–250.60 and B1–250M; measured vs. calculated*

ity. In a recent study,[14] experimental evidence was given, which clearly indicates a minor role of the bending the upper part of corrugations. It should be noted that our experimental evidence indicated that a relatively much longer bend in bottom part is needed than that employed in their study to effect a substantial gain in capacity. Anyhow, it appeared that all proprietary designs perform similar in this respect, which means that nowadays the columns containing conventional structured packings can be revamped successfully using their advanced counterparts. Certainly, the application of high capacity structured packings in new designs will lead to leaner columns.

Figures 2 and 3 also illustrate a rather good predictive accuracy of the Delft method. A complete list of working equations of this complex, but straightforward method for conventional corrugated sheet structured packings is given elsewhere.[15] It should be noted that the method proved to be capable of predicting the effects of packing modifications on the pressure drop and capacity, by adjusting appropriately geometry related parameters. On the mass transfer side; there was no need for special provisions in the model, because it was found that the efficiency deteriorates only slightly due to bending the bottom and/or upper part of corrugations.

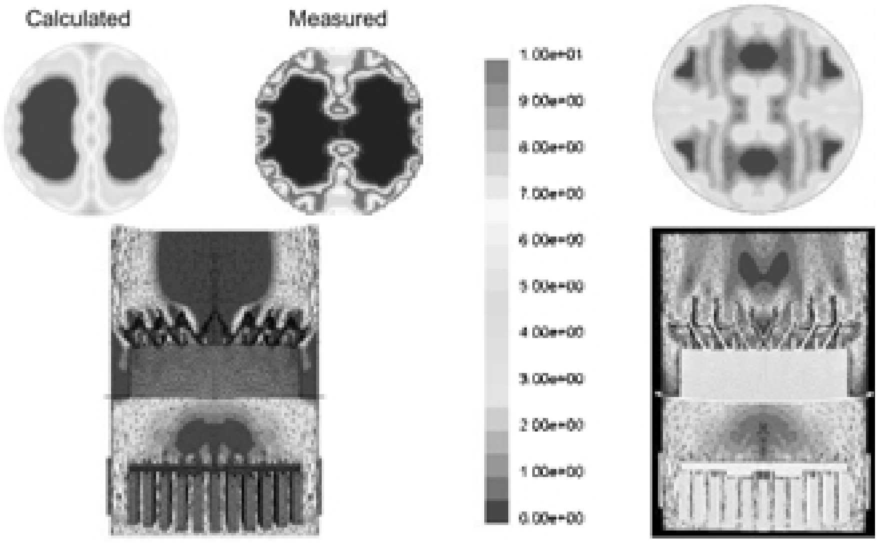
Further packing geometry development and optimisation work will be substantiated experimentally by using a new total reflux distillation column (internal diameter of 0.45 m, packed height up to 2.5 m) available now at TU Delft. Major pieces of equipment for this impressive installation (see the photograph shown in Figure 4) were donated by J. Montz.

CFD streamlined liquid collectors/gas redistributors

A potential drawback of further reduction in the pressure drop of a packed bed may be the reduction of the driving force for smoothing out efficiently the heavily maldistributed initial gas profiles generated by conventional liquid collectors installed in between packed beds. A comprehensive experimental and simulation study carried out at TU Delft[16–18] indicated that a state of the art Computational Fluid Dynamics (CFD) tool is capable of predicting reliably the effect of column internals geometry on the single-phase gas flow field. With this in mind, CFD has been used to improve the performance of common low pressure drop liquid collectors that, designed primarily to enable undisturbed collection and remixing of liquid proved to act as a kind of gas maldistribution generator.[18]

F i g . 4 – *Photograph of the TU Delft total reflux distillation column*

Figure 5 shows the CFD snap-shots of the gas flow field for the conventional and an improved configuration as well as the corresponding cross sectional velocity distribution patterns at the distance corresponding to the inlet to the bed above. The comparison of predicted and measured gas distribution profiles of the conventional (left hand side) liquid collector indicates that CFD simulation is capable of predicting reliably the gas distribution performance of this kind of packed column internals. A closer inspection of the CFD snap-shot shown on the left hand side of Fig. 5 indicates that in all cases a relatively narrow gas jet is passing along the tip of the opposite blade (side exposed to the liquid draining from the bed above). To avoid this, in new design, shown on the right hand side of Figure 5, the existing blades are extended and bended smoothly inwards to deflect the gas stream in the vertical direction. In addition, the central part, a chimney like design with two superimposed V-shaped covers, is changed into a simple, upside-down turned V-shape liquid collecting trough. The corresponding Cv (coefficient of variation) value is 58 %, which, compared to 95 % of the conventional design indicates a significant reduction in the magnitude of flow variation. This in combination with a more streamlined central part resulted in roughly a 28 % lower pressure drop (63 Pa). From the internals design/performance point of view, this CFD simulation result is quite encouraging. Namely, it indicates that increased pressure drop is not required to improve the initial gas distribution, i.e. that with respect to conventional designs, there is still a significant potential for improvement in both



F i g . 5 – *CFD snap-shots of a side cut of the gas flow through liquid redistribution section equipped with a conventional (left, below) and a modified (right, below) liquid collector; above, on the left hand side, measured and CFD predicted distribution profiles at a distance of about 0.35 m above collector are shown; on the right hand side the predicted profile for modified collector configuration is shown* the quality of the initial gas distribution and the associated pressure loss. A practical problem with the use of CFD is a rather long run time (up to 20 hours) associated with this kind of simulations.

Partially flooded packed beds

Major feature of another cooperative effort of Montz and BASF with the University of Karlsruhe (Prof. M. Kind) is concerned with the utilisation of the mass transfer enhancement potential of operating columns preferentially in loading range. To avoid danger of flooding the loading range operation should be controlled in a way. As presented in a paper by B. Kaibel et al.[19] this can be done by combining low and high specific surface area packings, i.e. by operating some kind of hybrid packed beds. This experimental study indicates that some 50 % of efficiency enhancement can be obtained (Iso/n-butanol system at 1.013 bar in a 0.1 m internal diameter test column) using such a configuration. Certainly such operation implies a higher specific pressure drop, however this is not at the cost of capacity. The problem lies merely in the fact that the range of enhanced performance operation appeared to be quite narrow and actually too close to flooding limit.

Better performance could be expected from a bed where standard packing elements are combined with shorter, specially designed elements to promote bubbling action similar to that of a tray. As indicated schematically in Figure 6, these bubbling promoters should be short enough to avoid deteriorating effect of liquid back mixing, i.e. to provide for a longer residence time for liquid, a larger interface and an intensive contact of two phases, allowing at the same time a smooth disengagement of vapour and liquid under high liquid load conditions. A first industrial application of partially flooded packings was reported most recently.[20] In this case these new packings were used to provide a longer residence time in a homogeneously catalysed reactive distillation in a column with a diameter of 0.7 m. So, a considerable progress along this line is made and it is expected that the total reflux experiments at larger scale could provide revealing answers with respect to hydraulic stability and the extent of mass transfer enhancement of partially flooded beds.

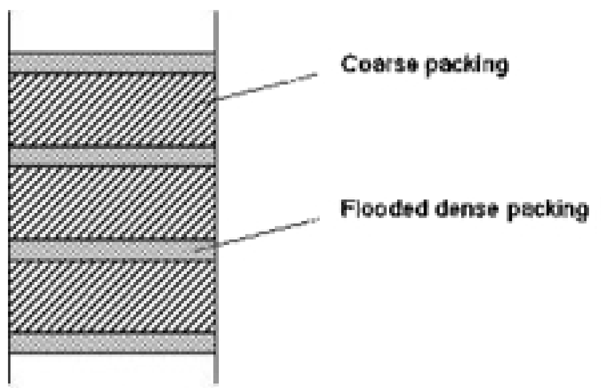


Fig. 6 – *Schematic representation of a hybrid packed bed containing high liquid load sections*

Catalytic structured packing

A cooperative effort with the Prof. A. Gorak's group from the University of Essen, now the University of Dortmund, led to the development and commercialisation of a hybrid type, catalytic structured packing (Montz Multipak), containing vertically oriented, segmental designed gauze material catalytic bags placed in between corrugated wire gauze sheets with alternatively oriented flow channels. A photograph and a front cut of a packing element of Multipak is shown in Figure 7. Two different designs are distinguished, the Multipak I with standard, 500 m^2/m^3 wire gauze corrugated sheets with an corrugation inclination angle of 60 degrees, and Multipak II comprising thicker catalyst bags (larger specific catalyst volume) sandwiched between the corrugated sheets of a 700 m^2/m^3 wire gauze packing with an corrugation inclination angle of 45 degrees. This provides some flexibility regarding balancing the reaction and separation requirements. Hydraulic and mass transfer performance of Multipak I as determined using pilot scale installations with different internal diameters and packed heights is described thoroughly elsewhere.[21–23]

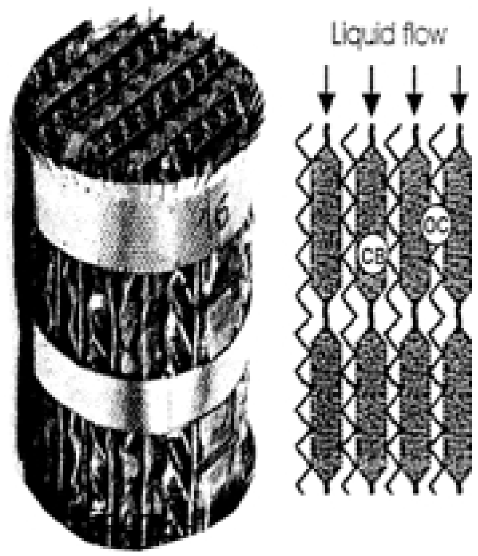


Fig. 7 – *Photograph of a laboratory-scale packing element of Multipak with the shematic illustration of internal configuration; CB denotes catalyst bags, OC corrugated sheets*

The trouble with reactive distillation is that due to excessive costs the experimentation with real systems is limited to too small columns, i.e. there is no intermediate/semi-industrial scale effort available to develop systematically the necessary scale-up knowledge. This is expected to be developed by combining model developments and non-reactive, i.e. hydraulics and separation efficiency related tests at larger scale to validate accordingly predictive models, taking into account reaction kinetics information obtained from small-scale experiments. This approach is outlined to some extent in ref. 23. A state of the art review of the reactive distillation technology based on BASF experiences is given in the paper by Althaus and Schoenmakers.[24] A special issue of Chemical Engineering and Processing (March 2003) is devoted to reactive separations.

Finally, it should be noted that in the meantime J. Montz GmbH decided to drop this line of development and transferred its rights for Multipak to Sulzer Chemtech. Based on a much wider involvement with all aspects of this technology, Sulzer Chemtech expects a breakthrough to happen in this field upon consolidation of the implementation related knowledge.[25]

Dividing wall column

First concepts of dividing wall (partitioned) columns can be traced back in 1930s and 1940s.[26,27] In the same period, Brugma[28] laid the fundaments for what is known today generally as thermally coupled distillation. However the credit for full recognition of the energy conservation potential of fully thermally coupled distillation columns goes to Felix Petlyuk, who published with co-workers in the mid 1960s a paper[29] that represents the milestone in the development of this technology. A dividing wall column (DWC) is the most daring variation of the so-called Petlyuk column. Its major feature is that it allows substantial energy savings, while separating in a single body a three-component mixture into pure products. A conventional configuration for separation of a three-component mixture into pure components is shown in Fig. 8 together with a Petlyuk- and a DWC configuration. Obviously, the dividing wall column represents the most compact configuration, which allows both considerable energy and capital saving. However, in spite of potential benefits, for years DWC remained to be an exotic academic concept and even the energy price explosion of mid 1970s was not good enough to push toward its industrial implementation. Certainly, it may look strange that the application pioneer, the company BASF, realised some twenty columns before other companies considered this as a possibility.

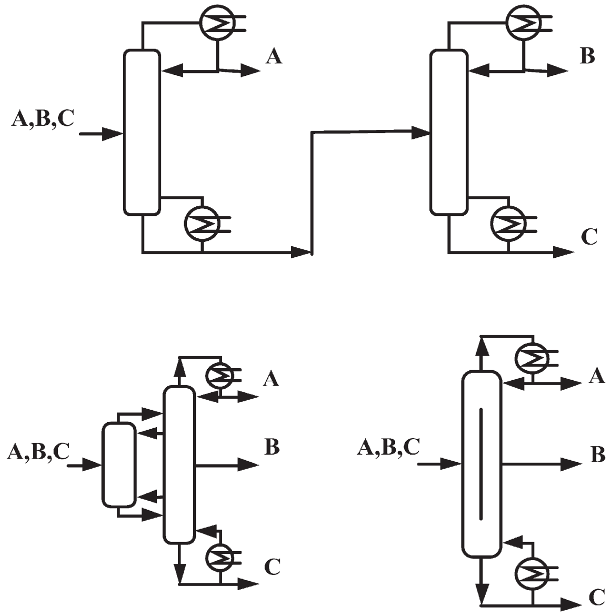
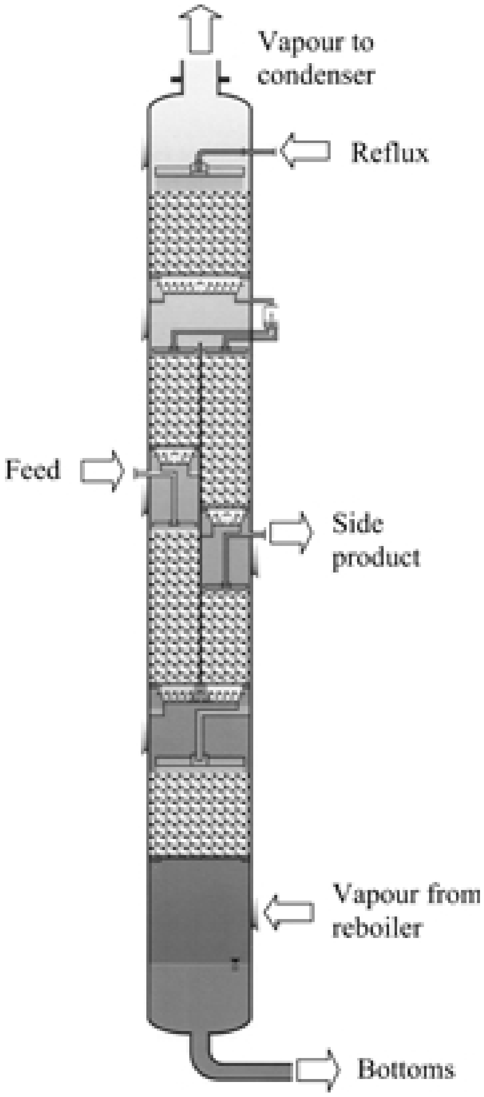


Fig. 8 – *Distillation configurations for separation of a three component mixture: (a) conventional, (b) thermally coupled – Petlyuk, and (c) dividing wall column*

Reintroduced to the distillation community by G. Kaibel in 1987,[30] the dividing wall column is considered today to be an established technology with a steadily growing application potential, including also separation of four component mixtures. Energy savings with respect to conventional two-column arrangements are in the range of 30 %. A realistic idea on this and other practical benefits from the utilisation of DWCs can be obtained from a paper by Ennenbach et al.[31] The most recent paper by Kolbe and Wenzel[32] discusses the potential for application in processing petrochemical cuts. Certainly academic people also embarked on this idea, and over the last ten years a good deal of academic and industrial research effort went into the evaluation of the energy saving potential in various applications.[33–35]

However it should be noted that the implementation of this concept required specific constructional solutions. An idea on the internal configuration of a packed DWC can be obtained from Figure 9. Special DWC related know-how was developed from the beginning at J. Montz GmbH, which built the BASF columns. For instance, the first columns were provided with a fixed dividing wall, which was welded on both sides to the column shell. To provide for better flexibility of the internal configuration and to reduce the design accuracy requirements of the system, a free, movable wall system was employed in new designs. For larger diameter columns the dividing wall is built by assembling it in the column from specially designed, easy to install manhole size segments (see Fig. 10). Such details including the edge seals and other constructive solutions belong to the proprietary know-how of Montz. Modern concepts



F i g . 9 – *Artist impression of a packed DWC*

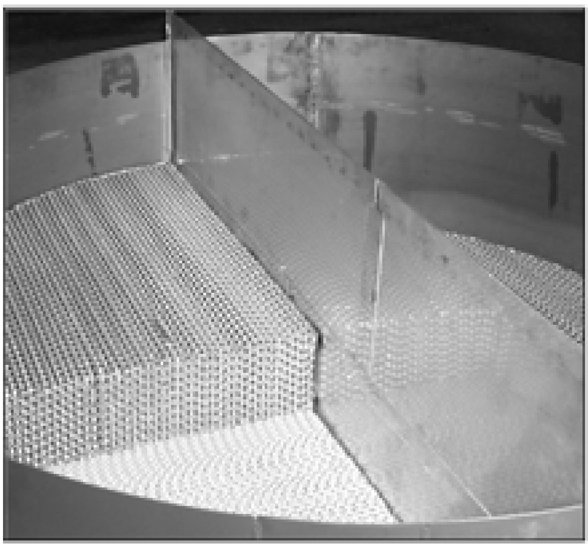
also provide off-centre positions of the dividing wall to meet special requirements, e.g. for vapour feeds in high vacuum applications. The dividing walls are preferably combined with special self-adjusting packings to avoid assembling problems.

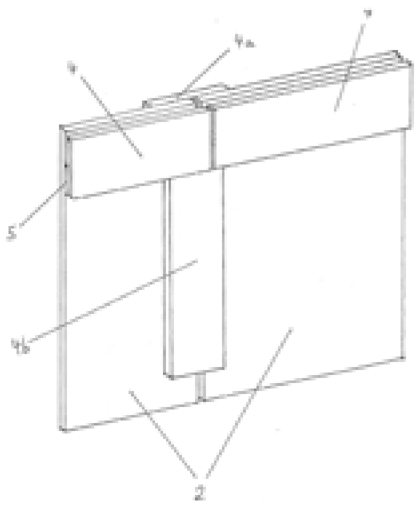
Certainly a considerable research effort proceeded industrial implementation. The necessary knowledge of hydrodynamics of DWC was collected during air/water and tracer testing on large scale (0.8 m inner diameter) carried out in Hilden in cooperation with TU Delft. These experiences helped to develop the full design and construction know-how that represents the basis for the realisation or much larger packed columns than in the beginning (diameters first below 1 m, now up to 4 m). With the increase in diameter it became possible to consider the use of trays in applications where trays offer advantages over packings. Meanwhile several tray DWCs with diameters up to 5 m are in operation. Certainly, the operation of a DWC requires a more advanced and elaborate control scheme. This aspect is for instance discussed by *Adrian* et al.[36]

The layout of a DWC can be made on the basis of computer simulations as in the case of conventional column arrangements. In cases where experiments have to be made laboratory scale columns with diameters down to 50 mm can be used (see Figure 11).

Concluding remarks

During the last ten years, J. Montz GmbH, an established worldwide operating medium size process equipment manufacturer managed in a number





F i g . 1 0 – *Photograph illustrating the assembling a packed column with proprietary loose dividing wall elements*

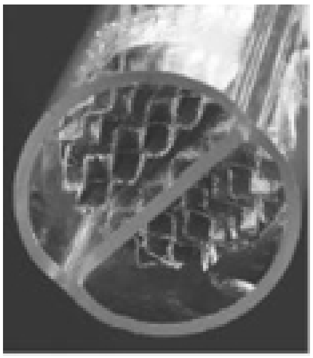


Fig. 11 – *Photograph of a segment of a mini plant (50 mm diameter) DWC*

of cooperative efforts with some German and a Dutch university as well as BASF to make technology advances which helped to preserve its share in ever harshening distillation equipment market.

Among others, the nature of pressure drop in a corrugated sheet structured packing has been revealed and manipulated accordingly. A significant improvement in capacity of a conventional corrugated sheet structured packing has been achieved: simply by bending smoothly to the vertical the corrugations in the lower third of a sheet. With respect to the performance of 60° packing, the capacity of M-series packing is still on the shorter side, indicating that the considerable pressure drop due to gas/gas interaction also influences the capacity. In the range of interest for application of this packing the efficiency matches that of the original packing.

Certainly, further packing performance improvement is possible, and optimising the liquid redistributor design will compensate for a loss of gas distribution capability of a bed due to reduced pressure drop. Progressing along this line we could eventually arrive at a solution that will approach closely the theoretical potential for this kind of contacting devices.

The Delft model proved to be versatile enough to account appropriately for all geometry manipulations considered in our packing improvement studies. Since it does not require any empirical, packing specific constant it enables a tailor made approach to the design and retrofitting of distillation columns containing corrugated sheet structured packing.

CFD appeared to predict well the single phase flow fields in configurations encountered in packed columns and the simulation tools like Fluent may be considered as a useful aid for design and evaluation of performance of packed column internals. Major difficulty, which may work adversely to potential users is the immense run time associated with CFD simulations. The simulation capability, regarding the real, two-phase flow situations is still in an early stage of development.

Hybrid packed beds with gas-liquid contacting enhancement means may prove useful, if not widely for separation purposes than certainly for catalytic distillation purposes. Hybrid catalytic elements proved to work in practice and the ongoing performance optimisation effort will result in technically and economically more elegant designs than it was the case with the industrial applications of the first generation of catalytic packings.

Dividing wall column is now considered to be an accepted technology and is expected to grow steadily in the number and variety of applications in industrial practice. This growth will accelerate further improvements in this technology where necessary.

In this time of globalisation of all aspects of human life, with less ground left for small undertakings, faster innovation cycles are an imperative. It is our belief that a more effective utilization of resources available within this proven, industrial users/university/equipment manufacturer collaborations formula will help us to respond successfully to challenges for faster technology developments in the field of distillation.

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