STRUCTURED PACKING FLOODING: ITS MEASUREMENT AND PREDICTION

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A cryogenic pilot distillation column operating at total reflux is described. It was used to measure the pressure drop and flooding of various structured packings, first using pure nitrogen and then using an oxygen-argon mixture. By varying the pressure, the nitrogen system was used to characterize the capacity of each packing via Wallis plots. The oxygen-argon flooding data can be accurately predicted only if it is assumed that the pressure drop at flooding is proportional to the liquid density, in agreement with an earlier suggestion by McNulty.

KEYWORDS: structured, packing, flooding

INTRODUCTION

Two flood points are important for structured packing. One is where the HETP starts to rise rapidly - the maximum operational capacity. Another is where the pressure drop starts to rise rapidly - the hydraulic flooding capacity. This paper deals with the latter.

Fundamental models to predict the pressure drop and flooding of structured packing have so far not been very successful. The regular arrangement of the crossing triangular flow channels has seduced many workers, mainly in universities, into believing that useful progress can be made via detailed fundamental modeling. As recent examples, Valluri et al. (1) modeled liquid film flow over structured packing and Petre et al. (2) modeled gas flow by CFD. In each case, only single-phase flow was considered. The extension of this type of work to consider two-phase flow at flow rates approaching flood conditions seems to be as far away as ever.

As a practical alternative, several semi-fundamental models have been proposed. For example, Olujic and his co-workers (3) developed a correlation for the so-called loading point that occurs typically at a pressure drop of about 1-2 mbar/m. They also gave an empirical correction to predict the pressure drop above the loading point. However, even though the model contains six regression coefficients, it is still not accurate enough to predict the flooding point with any confidence.

Over the past few years, it has become apparent that the critical region for flooding lies at the interface between packing bricks or sections, and not so much in the regions in the body of the bricks that are perhaps more easily modeled. At the interface, liquid has to drip or wick from the base of each sheet to the underlying sheets that cross it at 90° . This results in an increase in the local film thickness and a corresponding restriction in the area available for vapor flow. Effective models of pressure drop, liquid holdup and flooding will have to deal with this transition region. There are numerous new designs of "high

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capacity" packing that accommodate this transition in different ways, but all of them seek to reduce the velocity of the vapor in the transition region so as to allow the liquid to cross from one brick to the next with the minimum of upwards drag. Additionally, useful fundamental and empirical models must deal with: the geometry of the packing – crimp size, crimp angle, corrugation angle to the vertical, surface texture and sheet perforations; the wall reflection, both of vapor and liquid (as a function of column diameter), and the effect of wall wipers; and, of course, the physical properties of the vapor and liquid, including density, viscosity and surface tension.

As a result of these difficulties, practical design still makes use of the various general empirical models for pressure drop and flooding that have been proposed over the years. Most of them have been summarized by Kister (4). He deals extensively with the many forms of the generalized pressure drop correlation (GPDC) in which each packing is characterized by a single parameter – the packing factor. An alternative flooding model for structured packing is the Wallis plot, in which two parameters (the slope and intercept) are used to characterize each packing. It has been shown previously how the GPDC and the Wallis plot are related (5). In order to determine the necessary parameters for each packing, experimental data are necessary and it can be obtained in various ways.

The easiest way to characterize a packing with regard to pressure drop and flood point is to test it using air and water at ambient conditions. Such data is often provided in manufacturers' literature. However, there are a number of reservations about using air-water data to characterize structured packing, not least of which is the poor wetting of stainless steel by water because of its high surface tension. Bennett and Ludwig (6) have discussed this issue in some detail. A better approach is to test the packing using air and a liquid such as Isopar-M that has a surface tension of 26.6 dynes/cm at 25° C. Even so, problems remain because ambient air is not very representative of the vapor found in distillation columns. To overcome this, tests can be carried out in a pilot plant distillation column using the actual fluids that are of interest. Tests are normally carried out at total reflux, meaning that the liquid rate cannot be varied independently of the vapor rate. Operation of a pilot column at non-total reflux is fraught with inaccuracy, and is best avoided, because of the difficulty of measuring the internal flows within the column. In what follows we describe pilot plant tests at total reflux in which the liquid rate was varied independently of the vapor rate by operating the column at different pressures.

CRYOGENIC PILOT COLUMN

The pilot column for testing packings (and trays) under cryogenic conditions had an internal diameter of 305 mm and the packed height was typically 2.5 m. It operated at total reflux. A water bath surrounding the sump below the column provided the energy for boil-up. The water circulated through an external heater that controlled the bath temperature. The bath temperature and water bath height effectively controlled the boil-up rate. The vapor leaving the top of the packing was condensed in a shell and tube heat exchanger against liquid nitrogen that boiled and was vented to atmosphere. The pressure on the

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nitrogen (boiling) side of the condenser controlled the pressure inside the column. The distributor typically had 13 pour points and a riser height of 600 mm, so that it did not limit column capacity. A simplified sketch of the arrangement used is shown in Figure 1.

The internal liquid and vapor rates were determined from the measured power input to the water heater and a correction was made for heat leak into the column. The measured rate of addition of liquid nitrogen to the separator was used to check the energy balance. The measured pressure drop across the packing was corrected for hydrostatic head in the column and in the lines from the column to the differential pressure cell. So the reported pressure drops are due to flow only. The reported C_G and C_L values are those at the top of the packing.

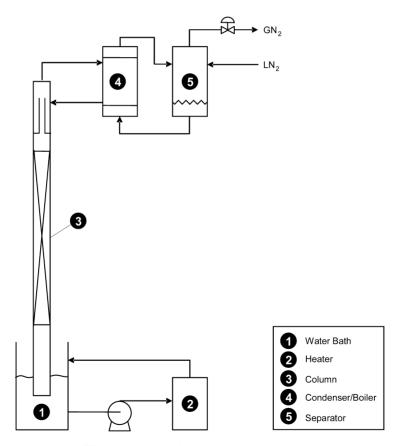


Figure 1. Cryogenic pilot column arrangement

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CHARACTERIZATION OF PACKINGS

Tests with pure nitrogen as the working fluid were used to characterize each packing. The column was operated at total reflux and at a pressure between 1.5 and 7.2 bar. The power input to the heater was adjusted until the pressure drop across the packing was 10 mbar/m. This pressure drop is just slightly less than the pressure drop at flooding while still allowing stable column operation to be maintained. Some other comparable values of the pressure drop at flooding used by other workers are:

Sulzer (7)	12 mbar/m
Kister and Gill (4)	
Mellapak 500Y	11 mbar/m
Mellapak 250Y	8 mbar/m
McNulty and Hsieh (8), Flexipac 1-4	16 mbar/m
Rocha, Bravo and Fair (9)	10 mbar/m

It is worth noting that some modern "high capacity" structured packings can operate at much higher pressure drops before flooding starts. For example, Billingham et al. (10) describe experiments in which flooding did not occur until the pressure drop approached 20 mbar/m.

The experimental results were plotted on a Wallis plot (11) using the equation:

$$C_G^{0.5} + m C_L^{0.5} = C \tag{1}$$

where

$$C_{\rm G} = \frac{M_{\rm G}}{\rho_{\rm G} A} \left(\frac{\rho_{\rm G}}{\rho_{\rm L} - \rho_{\rm G}}\right)^{0.5} \tag{2}$$

and

$$C_{\rm L} = \frac{M_{\rm L}}{\rho_{\rm L} A} \left(\frac{\rho_{\rm L}}{\rho_{\rm L} - \rho_{\rm G}}\right)^{0.5} \tag{3}$$

For a particular value of $C_{\rm G},$ the corresponding value of $C_{\rm L}$ is obtained at total reflux from

$$C_{\rm L} = C_{\rm G} \left(\frac{\rho_{\rm G}}{\rho_{\rm L}}\right)^{0.5} \tag{4}$$

Since the vapor density varies significantly with pressure, equation (4) shows that by operating at different pressures the ratio of C_L to C_G can be altered and a flood point locus for the packing may be generated.

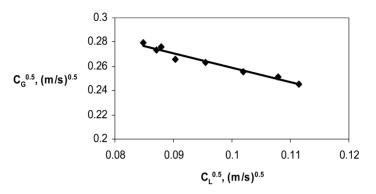


Figure 2. Wallis plot for Flexipac Type 1 using cryogenic nitrogen

A typical Wallis plot is shown in Figure 2. A wide range of packings were tested in this way, with specific surface areas ranging from 400 to $1000 \text{ m}^2/\text{m}^3$, corrugation angles to the horizontal ranging from 40 to 50° , crimp angles from 80 to 100° , surface textures from smooth to very rough, and with and without perforations. In addition, a number of "high capacity" packings were also tested that had crimp modifications at the interface between bricks. For each packing a Wallis plot was obtained and the packing was characterized by the parameters m and C.

A summary of the results obtained is shown in Figure 3. Also included on the figure are results reported earlier by Lockett (5) and McNulty and Hseih (8) obtained from

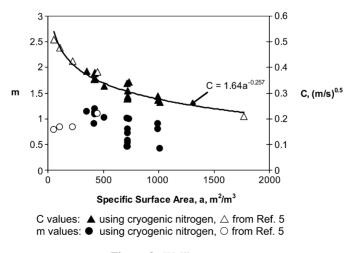


Figure 3. Wallis constants

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air-water tests. An interesting finding is that the parameter C varies as $a^{-0.257}$, where a is the specific surface area. It has been previously shown (5) that C should be proportional to $a^{-0.25}$ from dimensional considerations of equation (1), and these results provide ample confirmation of this over a very wide range of packing variables. This finding is very strong evidence that the Wallis plot is the correct way to correlate structured packing capacity data. The variable m is less easily correlated. It appears that the variable m captures the effect of nuances in the details of the packing geometry as they influence packing capacity.

INFLUENCE OF LIQUID DENSITY ON THE PRESSURE DROP AT FLOODING

For each packing, additional tests were carried out with a cryogenic mixture of oxygen and argon. Both the pressure drop and HETP were measured with this system and the column was operated at total reflux. For all tests, the composition of oxygen in the vapor at the bottom of the packing was maintained at 95% molar and the column pressure was 1.5 bar. Some of the HETP results have been reported elsewhere (12).

In Figure 4, the experimentally determined values of C_G that resulted in a pressure drop of 10 mbar/m in the oxygen-argon tests are compared with the predicted values of C_G for each packing using the values of m and C obtained from the nitrogen tests. The figure shows that the measured C_G falls short of the predicted C_G by about 6%.

The discrepancy is attributed to the use of a single pressure drop (10 mbar/m) to characterize the flood point for the two systems. Some previous workers, notably Prahl (13), Robbins (14), and McNulty (15) have pointed out that the Generalized Pressure Drop Correlation (GPDC) has problems at very low values of the flow parameter. The liquid loading is very small as the flow parameter approaches zero and so the predicted pressure drop should be close to the dry pressure drop. However, the GPDC indicates

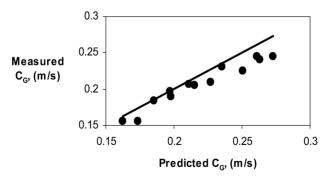


Figure 4. Measured C_G for O_2 -Ar at $\Delta P = 10 \text{ mbar/m}$ versus C_G predicted from Wallis constants obtained from N_2 results

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that the dry pressure drop depends on the liquid density, which is clearly unreasonable. To overcome this problem McNulty (15) proposed a revised version of the GPDC in which the pressure drop at flooding was a function of liquid density.

Assuming that the dry pressure drop is small compared with the total pressure drop, McNulty argued that the pressure drop at flooding can be considered as the force opposing the gravitational force on the liquid holdup. Hence,

$$\Delta P_{\rm F} = h_{\rm F} (\rho_{\rm L} - \rho_{\rm G}) g \tag{5}$$

where ΔP_F is the pressure drop at flooding and h_F is the liquid holdup at flooding.

If the vapor density is small compared with the liquid density and the liquid holdup at flooding is the same for different systems, it follows that,

$$\Delta P_F \propto \rho_L \tag{6}$$

In the present work at the flood point, the dry pressure drop was estimated to be 12% and 27% of the total pressure drop for the nitrogen and oxygen-argon systems, respectively, which is in reasonable agreement with McNulty's assumption. These estimates were obtained from Sulpak (16) for Mellapak 750Y.

To accurately estimate the effective liquid density for the oxygen-argon system an integrated value over the column should be used. However, in this case we decided to use simply the density of a 50% molar mixture which is 1233 kg/m³. The mean composition over the column height was typically about 50%. The liquid density for nitrogen was determined at a mean pressure of 3.7 bar and is 744 kg/m³. Using these values in equation (6), with $\Delta P_F = 10 \text{ mbar/m}$ for nitrogen, gives $\Delta P_F = 10 \times 1233/744 = 16.6 \text{ mbar/m}$ for oxygen-argon.

Figure 5 shows the experimentally determined values of C_G that give a pressure drop of 16.6 mbar/m in the oxygen-argon tests, compared with the predicted values using the values of m and C from the nitrogen tests.

Clearly, the over-prediction is removed and the predicted flooding results for oxygen-argon agree very well with the measured values. The conclusion is that these results confirm McNulty's suggestion. The pressure drop at flooding indeed appears to be different for each system and to be proportional to the liquid density. This finding will have its greatest importance when the liquid density of the fluid used to characterize the packing is very different from the liquid density of the fluid for which predictions are needed. The use of air-water data to predict flooding of distillation systems is an example where there could be a significant error involved because of this.

CONCLUSION

It has been shown how a pilot distillation column operated at total reflux and at different pressures can be used to characterize the flooding behavior of structured packing using a

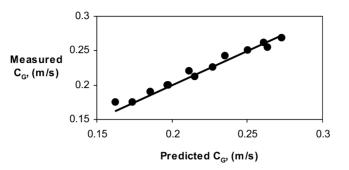


Figure 5. Measured C_G for O_2 -Ar at $\Delta P = 16.6$ mbar/m versus C_G predicted from Wallis constants obtained from N_2 results

Wallis plot. The results support the contention that the pressure drop at flooding is proportional to the liquid density as suggested earlier by McNulty.

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NOMENCLATURE

А	Column cross sectional area, m ²
а	Specific surface area, m^2/m^3
С	Constant, $(m/s)^{0.5}$
C _G	Capacity factor for vapor, m/s
C _L	Capacity factor for liquid, m/s
g	Acceleration due to gravity, m/s^2
h _F	Fractional liquid holdup at flooding
M _G	Vapor mass flowrate, kg/s
M_L	Liquid mass flowrate, kg/s
m	Constant
ΔP	Pressure drop per unit bed height, mbar/m
$\Delta P_{\rm F}$	Pressure drop per unit bed height at flooding, Pa/m
$ ho_{ m G}, ho_{ m L}$	Vapor and liquid densities, kg/m^3

REFERENCES

- 1. Valluri, P., Matar, O.K., Hewitt, G.F. and Mendes, M.A., 2005, Thin film flow over structured packings at moderate Reynolds numbers, *Chem. Eng. Sci.*, 60: 1965.
- 2. Petre, C.F., Larachi, F., Iliuta, I. and Grandjean, B.P.A., 2003, "Pressure drop through structured packings: Breakdown into the contributing mechanisms by CFD modeling, *Chem. Eng. Sci.*, 58: 163.
- 3. Verschoof, H., Olujic, Z. and Fair, J.R., 1999, A general correlation for predicting the loading point of corrugated sheet structured packings, *Ind. Eng. Chem. Res.*, 38: 3663.

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- 4. Kister, H.Z., 1992, Distillation Design, McGraw-Hill, New York.
- 5. Lockett, M.J., 1995, Flooding of rotating structured packing and its application to conventional packed columns, *Trans. IChemE*, 73A: 379.
- 6. Bennett, D.L. and Ludwig, K.A., 1994, Understanding the limitations of air/water testing of distillation equipment, *Chem. Eng. Prog.*, April, 72.
- 7. Sulzer Brothers Ltd., 1991, Separation columns for distillation and absorption, Winterthur, Switzerland.
- McNulty, K.J. and Hsieh, C., 1982, Hydraulic performance and efficiency of Koch Flexipac structured packings, *AIChE Annual Meeting*, Los Angeles, Nov. 14–19.
- 9. Rocha, J.A., Bravo, J.L. and Fair, J.R., 1993, Distillation columns containing structured packings: A comprehensive model for their performance. I. Hydraulic models, *Ind. Eng. Chem. Res.*, 32: 641.
- Billingham, J.F., Seiler, D.M. and Lockett, M.J., 1999, Method for operating a cryogenic rectification column, US Patent 5,921,109.
- 11. Wallis, G.B., 1969, One-dimensional two-phase flow, McGraw Hill, New York.
- Lockett, M.J., 2004, Cryogenic distillation: Past and present innovations, Proc. First China-USA Joint Conference on Distillation Technology, Tianjin, China, July 15–17: 324.
- 13. Prahl, W.H., 1970, Liquid density distorts packed column correlation, *Chem. Eng.*, 77 (24): 109.
- Robbins, L.A., 1991, Improve pressure drop prediction with a new correlation, *Chem. Eng. Prog.*, May, 87.
- 15. McNulty, K. J., 1999, What's wrong with the GPDC, AIChE Meeting, Dallas, November.
- 16. Sulpak 2.1, 1999, Design Program, Sulzer Chemtech, Winterthur, Switzerland.