SYMPOSIUM SERIES NO. 152

EXPERIMENTAL INVESTIGATION OF REACTIVE DISTILLATION PACKING KATAPAK® - SP 11: HYDRODYNAMIC ASPECTS AND SIZE EFFECTS

Elisabetta Brunazzi* and Aurora Viva
Laboratory of Process Equipment, Department of Chemical Engineering, Industrial Chemistry and Material Science, University of Pisa, via Diotisalvi 2, 56126 Pisa, Italy,
*E-mail: e.brunazzi@ing.unipi.it

This paper presents the results of an experimental study carried out to examine the hydraulic behaviour of Katapak®-SP11, a new structured catalytic packing of Sulzer Chemtech. Liquid holdup, dry and wet pressure drop, gas capacity and loading of two sizes of packing have been measured using the air-water system at ambient conditions. The experimental results collected are useful for supporting theoretical developments in hydrodynamic modelling and analysis of scale up issues. Data have been used to derive a set of correlation equations to describe the above quantities in terms of gas and liquid loads.

KEYWORDS: catalytic packing, fluid dynamics, Katapak®, structured packing, reactive distillation, liquid holdup

INTRODUCTION

The possibility of exploiting an advantageous interaction between separation and reactive operations has favoured the implementation of heterogeneous catalysis in reactive distillation columns. Catalytic internals have therefore to satisfy at the same time the needs of chemical reaction, distillation and capacity. To meet this requirements, structured catalytic packings have been developed, which combine the advantages of a structured packing with the features of a catalyst support [1]. The hardware and the design have become priority aspects of this integrated concept, and hence adequate knowledge and understanding of the hydrodynamics occurring in this novel internals and of their mass- and heat-transfer behaviour are of fundamental importance [2, 3].

In this work, the hydrodynamic aspects of KATAPAK®-SP 11, a novel structured catalytic packing of Sulzer Chemtech, were investigated. Liquid holdup, dry and wet pressure drop, gas capacity and loading of two sizes of packing have been measured using the air-water system at ambient conditions. The results provide useful information for supporting the development of reliable simulation tools, for instance through the rate based models, and the design of reactive separation columns.

PACKING AND COLUMNS DESCRIPTION

The structure of KATAPAK-SP® (SP for Separation Performance) consists of "separation elements" (Layers of Mellapak or MellapakPlus packings) and "reaction elements" (wire-gauze catalyst bags filled with catalyst particles of about 0.8 to 3 mm in size) assembled in

alternate sequence. The layers are held together by wire gauze collars, whose peculiar shape is also used to pour the liquid flowing along the wall back inside the separation and reactive zones. Thanks to its modular design, KATAPAK®-SP can be used for different reaction system, varying in the best proportion the catalyst and the separation fractions. In particular, KATAPAK®-SP type 11 combines one layer of corrugated sheet, with specific surface area of 500 m^2/m^3, per catalyst bag and provides a catalyst volume fraction up to 40% for the larger packing diameters [1]. To gain insight into scale up aspect, in this work KATAPAK®-SP11 has been used in two different sizes (Figure 1). In particular KATAPAK®-SP 11 with nominal diameters of 50 and 100 mm, fitted in test column C50 (50 mm ID) and C100 (100 mm ID), respectively, have been characterised. First, a geometrical characterisation of the two packings was performed with the goal of determining fundamental characteristics among which the void fraction and the specific superficial area. The main differences between the two packings are pointed out in Table 1, where also values for the Mellapak types packing are listed. The specific superficial area, a, has been evaluated considering the contribution of Mellapak, based on the volumetric fraction taken by the Layers, and that of Catalyst Bags, coincident with the area of the external wire gauze of the catalyst bags. The values of void fraction, ε_{VF}, derived from the geometrical characterisation are in good agreement with the measured values obtained resorting to Archimedes' principle.

Test Columns made of plexiglass have a total bed height of 2 m. Due to the different heights of the packing elements, C50 was filled with 20 elements of KATAPAK®-SP 11 DN50, whereas 10 elements of KATAPAK-SP 11 DN100 were used in C100. Consecutive packing elements are rotated relative to one another at an angle of 90°. Being this work directed to the hydrodynamic study of the packing and not to the reactive behaviour, the catalyst bags were filled with glass spheres with 1 mm of diameter.

Figure 1. Katapak®-SP 11 elements with nominal diameters of 100 and 50 mm

Table 1. Characterisation parameters (KSP 11 = Katapak-SP11; MP752Y = MellapakPlus 752Y, M500Y = Mellapak 500Y, M250Y = Mellapak 250Y)

	KSP11–DN 50	KSP11–DN 100	M500Y/MP752Y	M250Y
H (mm)	100	200	200	200
a (m^2/m^3)	199	203	500	250
ε_{VF}	0.81	0.767	0.975	0.975
$\varepsilon_{VF,\ measured}$	0.82	0.74	–	–
N° Layers	3*	5	–#	–#
N° Catalyst bags	2	4	–	–

Note: *Layers are of Mellapak 500Y instead of Mellapak Plus 752Y. #Number of layer depends on the diameter of the packing element

EXPERIMENTAL DEVICES

Experiments were carried out using the air-water system at ambient conditions. The experimental setup is presented in Figure 2 for the C100 device, being the scheme for C50 essentially the same.

The liquid is pumped to the top of the column and distributed over the packings by an appositively designed distributor, suitable for use over a wide range of liquid loads.

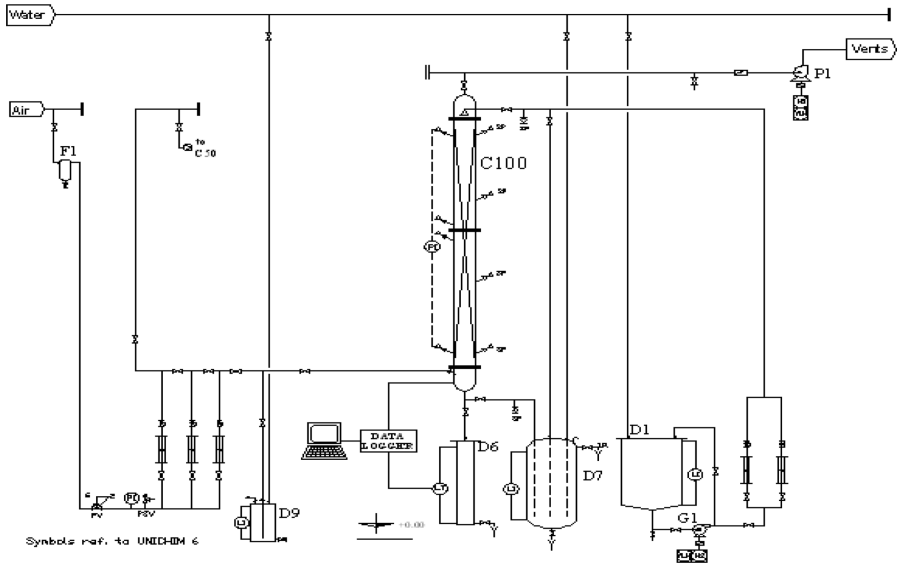

Figure 2. Pilot plant scheme

A compressor supplies air at flow-rates of up to 280 m^3/h at standard conditions. The gas flow-rate is set by means of valves and measured by flow-meters; pressure drops across the packing were measured by an inclined U-tube type manometer.

The dynamic holdup was measured using the volumetric method, i.e. measuring the amount of liquid draining from the packing after stopping the liquid feed. In order to determine quantitatively the amount per unit time of dropping liquid, a DP-cell was used to read the level in an purposely built liquid collecting tank. The signal from the DP-cell was acquired by a Data logger using a data rate of 2 readings per second and stored on a PC. Acquisitions lasted at least 1 h. The hold up was calculated as the volume of the liquid divided by the volume of the column (the part filled with the packing). Liquid loads up to 30 and 53 $m^3/h/m^2$were used for C100 and C50, respectively. Gas loads up to flooding conditions were employed for both columns.

RESULTS AND DISCUSSION

DRY PRESSURE DROP

Dry pressure drop were measured across the packing when only air flowed upwards through the Test Columns. The results were correlated in terms of the gas F-factor, F_V, by means of the following equation:

$$\frac{\Delta P_{Dry}}{\Delta z} = a \cdot F_v^b$$

with coefficient a equal to 149.13 and 136.37, and coefficient b equal to 1.823 and 1.671, for columns C50 and C100, respectively. This relation provides a correspondence with experimental data with very high precision ($R^2 > 0.99$).

WET PRESSURE DROP

The experimental data obtained for the irrigated packing have been analysed considering the trend of the pressure drop $\Delta P_{wet}/\Delta Z$ versus gas F-factor at constant specific liquid loads, L, in a double logarithmic representation. For each liquid flow rate, two regions are pointed out, divided by the gas loading point. Below this point, the wet pressure drop is parallel to the dry pressure drop straight line, whereas above the loading point the wet pressure drop increases sharply, reaching the flooding (100%-capacity).

At a constant gas load, the influence of the liquid load on the pressure drop is not immediately recognizable. Ellenberger and Krishna [4] have observed that with the Katapak-S packing the wet pressure drop do not significantly increase with respect to the dry pressure drop until a certain critical value of liquid load is reached. This behaviour has been explained considering that, due to the specific geometry of this packing, the liquid flows only inside the catalyst bags until the liquid loading point is reached. The liquid load point corresponds to the filling up of the catalyst bags. Therefore, at low liquid loads the wet pressure drop are determined only by the gas flowing in the

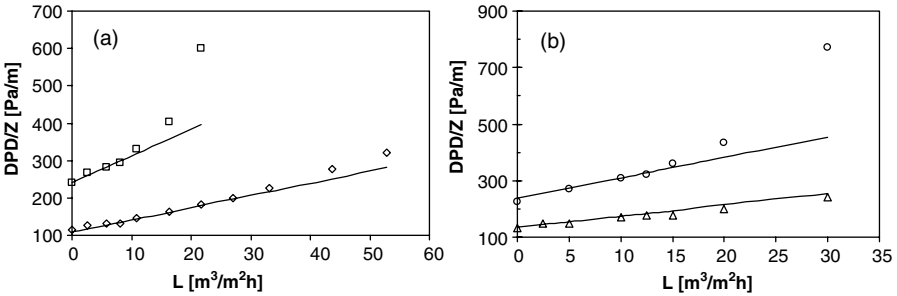

Figure 3. Wet pressure drop versus liquid load. a) C50. Gas friction factor: (□) 1.33 $Pa^{0.5}$, (◇) 0.83 $Pa^{0.5}$. b) C100. Gas friction factor: (○) 1.40 $Pa^{0.5}$, (Δ) 0.99 $Pa^{0.5}$

open channels. With Katapak-SP, on the contrary, the data obtained in the current work indicate that even at low liquid loads there is some liquid flow in the Mellapak layers contributing to the increase in wet pressure drop. In Figure 3 the experimental pressure drop versus liquid flow rates at different values of the gas friction factors are shown for the two columns. From the analysis of these data, it is clear that below the gas loading the wet pressure drop can be represented by a linear function of the liquid load. For both C50 and C100, the wet pressure drop is correlated in terms of liquid load and dry pressure drop as:

$$\frac{\Delta P_{Wet}}{\Delta z} = \frac{\Delta P_{Dry}}{\Delta z} \cdot (1 + 0.03 \cdot L)$$

This correlation has been validated below the gas loading in the operative liquid load ranges (From 0 to 30 m^3/m^2h for C100 and from 0 to 52.9 m^3/m^2h for C50). An impression of the overall accuracy of the correlation can be obtained from the parity plot shown in Figure 4. About 98% of the points fall within $\pm 10\%$, and the average absolute relative deviation is calculated as 5.9%.

LOADING AND CAPACITY

The gas capacity of the packing is limited by the onset of flooding. Even though the flooding point may be defined in different ways and its measurement is very difficult, from the experimental data it is possible to identify the gas load and hence the F-factor at which the slope of the pressure drop curves, at a given liquid load, go to infinity. The capacity can be represented in form of a Wallis-type diagram [5, 6]. This is a plot of $c_G^{0.5}$ versus $c_L^{0.5}$, where

- $c_G = \dfrac{F_v}{(\rho_L - \rho_G)^{0.5}}$ is the gas capacity factor;
- $c_L = \dfrac{L}{3600} \cdot \left(\dfrac{\rho_L}{\rho_L - \rho_G}\right)^{0.5}$ is the liquid capacity factor;

ρ_L and ρ_G are the liquid and the gas densities, respectively.

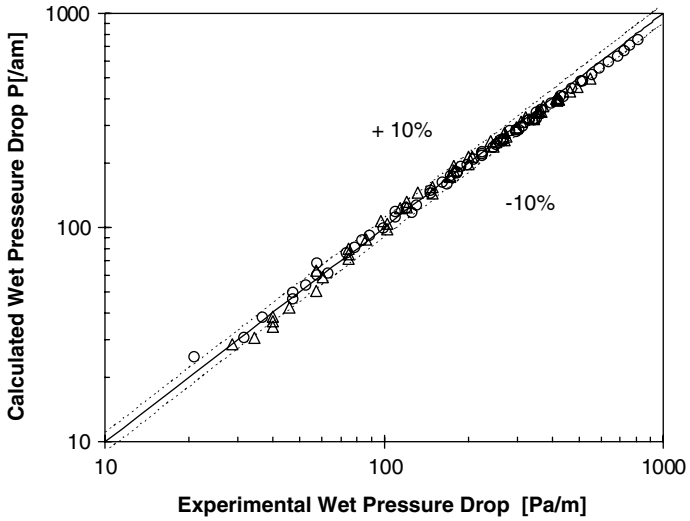

Figure 4. Calculated vs measured pressure drop for test columns. (○) C50; (Δ) C100

Examination of the experimental data obtained with the two sizes of packing has confirmed that also for this kind of packing the capacity, and thus flooding, can be represented through a linear relation between $c_G^{0.5}$ and $c_L^{0.5}$:

$$c_G^{0.5} + m \cdot c_L^{0.5} = c$$

where m and c are adjustable constants.

Moreover, it has been verified that similarly the gas loading can be correlated by a straight line in the Wallis Diagram. As already mention in section 4.2, the gas loading points are defined as points of operation where, in a double logarithmic representation of pressure drop versus F-factor, the deviation from the straight lines is observed. This study has pointed out that the slopes of both the capacity and the loading lines are equal, that is both lines are parallel:

$$c_G^{0.5} + m \cdot c_L^{0.5} = c_{Flood} \quad \text{Flooding Line}$$
$$c_G^{0.5} + m \cdot c_L^{0.5} = c_{Load} \quad \text{Loading Line}$$

where $m = 1.15$, $c_{Flood} = 0.307\ (\mathrm{m/s})^{0.5}$ and $c_{Load} = 0.286\ (\mathrm{m/s})^{0.5}$. The average absolute relative deviation is calculated as 2.0 and 2.6%, for the flooding and the loading lines

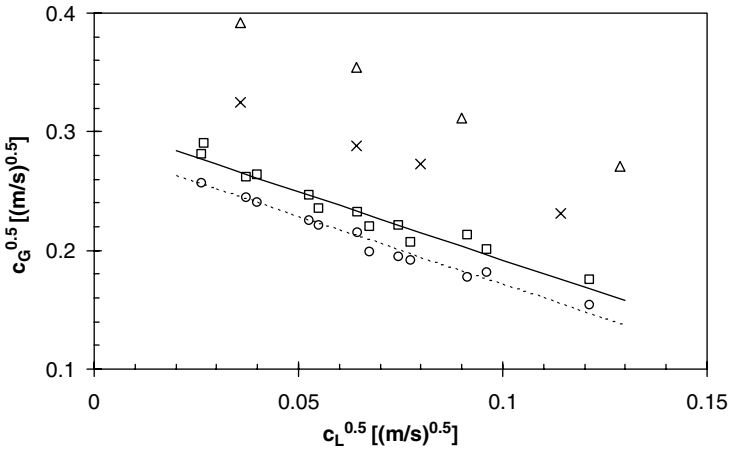

Figure 5. Capacity and loading data and correlations. Katapak-SP 11 DN 50 e DN 100: (○) Loading experimental data; (□) Flooding experimental points; solid lines: Correlations. Mellapak 500Y: (**x**) Flooding experimental data. Mellapak 250Y: (Δ) Flooding experimental data

respectively. It is also worth pointing out that according to Wallis the slope m is close to unity.

The capacity and the loading experimental data together with the correlation lines are presented in the Wallis Diagram in Figure 5. For comparison, capacity data for Mellapak 500Y and for Mellapak 250Y are also shown [6]. Figure 5 clearly shows that the different geometry of Mellapak and Katapak-SP 11 influences the onset of flooding. As a matter of fact, the presence of the Catalyst Bags causes an increase of the effective gas velocity and, consequently, the anticipation of the flooding.

DYNAMIC HOLDUP

The liquid holdup is an important parameter determining the packed tower pressure drop, capacity as well as efficiency [7]. Due to the geometrical complexity of Katapak-SP packing, particular attention has to be paid to chose an adequate draining time when using the volumetric method. Figure 6 shows the relative error $E(h_d)$ that can be made considering the amount of liquid dropping out of the packings after a time t, $h_d(t)$, with respect to the amount that is measured after 1 hour $h_d(1h)$, considered as the stationary value:

$$E(h_d) = \frac{h_d(1h) - h_d(t)}{h_d(1h)}$$

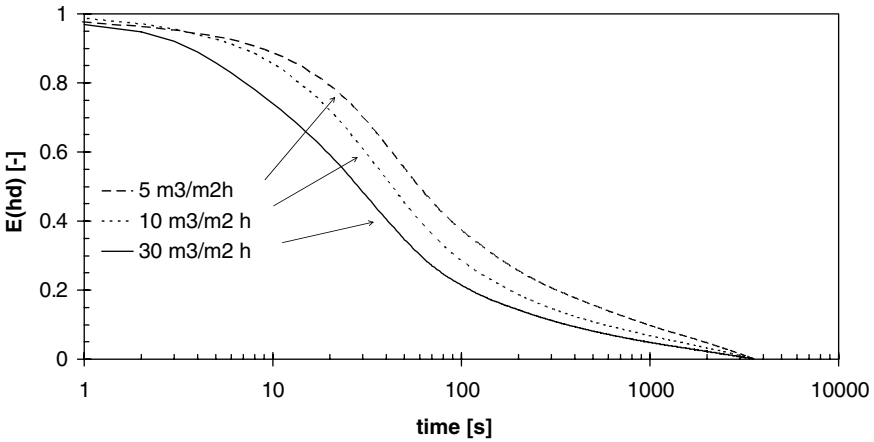

Figure 6. Relative error of dynamic hold up versus time at different liquid loads, for C100 measurements

Table 2. Coefficients of dynamic hold up correlation for test columns

d	*e*	*L*(*up to*)	
C50	0.0453	0.274	52.9
C100	3.2211	0.429	20.0

Larger errors are obtained with decreasing liquid loads. The values of dynamic liquid hold-up obtained after $1h$ drainage from stopping the liquid flow, $h_d(1h)$, were correlated as a power function of the liquid load $L(R^2 > 0.99)$:

$$h_d(1h) = d \cdot L^e$$

Coefficients d and e are given in Table 2.

CONCLUSIONS

Pressure drop, capacity and liquid holdup of two sizes of Katapak SP11 packing have been measured using the air-water system. Two sizes of packing were tested. From these data a correlation was derived to calculate the wet pressure below the gas loading point as a function of liquid load. Also, correlations were derived to identify gas loading point as well as flooding point as a function of liquid load. Dynamic liquid holdup values were measured

using the volumetric method. In particular, the amount per unit of time of dropping liquid was monitored by means of a DP-Cell for a duration of at least one hour. The results highlighted the importance to chose an adequate draining time.

The experimental results of this work will be useful for supporting theoretical developments in hydrodynamic modelling and analysis of scale up issues.

ACKNOWLEDGEMENT

We acknowledge the financial support provided by the European Commission within the 6th Framework Programme, Project "Insert – Integrating Separation and Reaction Technologies", Contract-No: NMP2-CT-2003-505862. The collaboration of Mr. Fabio Di Natale and Mr. Alessandro Rossi in carrying out the experimental program is gratefully acknowledged.

NOMENCLATURE

a	Specific superficial area of the packing, (m^2/m^3)
a, b	Coefficients of the dry pressure drop correlation, SI units
c_G, c_L	Gas and liquid capacity factors, (m/s)
c_{Flood}	Flooding constant, $(m/s)^{0.5}$
c_{Load}	Loading constant, $(m/s)^{0.5}$
d, e	Coefficients of dynamic holdup correlation
F_v	F factor, $(Pa^{0.5})$
$h_d(t)$, $h_d(1h)$	Dynamic hold up at time t and after 1 hour
H	Height of the packing element, (mm)
L	Specific liquid load, $(m^3/m^2/h)$ or (lt/h)
m	Slope of flooding and loading correlations
$\Delta P_{Dry}/\Delta Z$	Dry pressure drop, (Pa/m)
$\Delta P_{Wet}/\Delta Z$	Wet pressure drop, (Pa/m)
ε_{VF}	Void fraction of the packing from geometrical characterisation
$\varepsilon_{VF,\ measured}$	Measured void fraction of the packing
ρ_G, ρ_L	Gas and liquid densities, (kg/m^3)

REFERENCES

1. Goetze L., Bailer O., Moritz P., von Scala C., Cat. Today, 201, 2001
2. Kolodziej A., Jaroszynski M., Bylica I., Chem. Eng. Process., 457, 2004
3. Hoffmann A., Noeres C., Gorak A., Chem. Eng. Process. 383, 2004
4. Ellenberger J. and Krishna R., Chem. Eng. Sci. 1339, 1999
5. Spiegel L. and Meier W., IChemE Symp. Series No. 104, A203, 1987
6. Spiegel L. and Meier W., IChemE Symp. Series No. 128, 1992
7. Brunazzi E. and Paglianti A., AIChE J. 43, 317, 1997